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Countercurrent Rinsing on a High-Speed Halogen Tinplating Line

National Steel Corp, Weirton, W Va Weirton Steel Div

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COUNTERCURRENT RINSING ON A HIGH-SPEED HALOGEN TINPLATING LINE

by

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ABSTRACT

Countercurrent rinsing, as applied to high speed strip plating lines, involves the use of a compartmentised rinsing tank. The objective of the use of this method of rinsing is to reduce the amount of water required so as to have a volume of liquid more easily handled to recover the chemicals.

This report covers the first use of this type of rinsing on a high speed plating line. The first unknown to be studied was the operating performance of the multistage rinse system to determine whether or not the basic principles of countercurrent rinsing would hold for a high speed strip plating operation. Secondly, the best manner for recovering the chemicals in the concentrated stream from this rinse system had to be determined.

Efforts to recycle the concentrated rinse back into the main plating system and the problems which were encountered are also described in this report. New technology for solving these problems is described as well as an alternate method involving indirect recycling by means of the Detinning Plant.

This report was submitted in fulfillment of Grant (801982) received by the National Steel Corporation, Weirton Steel Division from the Environmental Protection Agency.

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I. INTRODUCTION

Weirton Steel Division of National Steel Corporation has completed research on the project "Demonstration and Evaluation of Countercurrent Rinsing for Reducing Pollution from a Halogen Tin Plating Line". This research was sponsored in part by the U.S. Environmental Protection Agency.

This project was designed to demonstrate that the use of counter-current rinsing is a viable approach to reducing pollution from this type of plating operation and to conserve valuable materials. This demonstration according to Environmental Protection Agency is an essential prerequisite to the industry-wide acceptance of this process because of the complexity of the electrolytic solutions used and the high speed at which the plating lines are operated. Any increase in the concentration of either the ionic constituents of the plating bath or the solid (sludge) material contained therein caused by the return of the concentrated effluent from the first rinse tank to the main electrolyte could result in either the production of poor quality or unsalesable tinplate or the frequent disposal of the expensive plating solution or both.

The demonstration and eventual application of countercurrent rinsing systems on halogen tin plating operations could have a significant impact on the amount of pollutants discharged from tin plating operations as approximately fifty percent of the tinplate in the United States is produced by the halogen process. In view of the above the project was initiated and completed in 16 months.

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Technical Background

In the halogen tinplating process, a steel strip is cleaned in an alkaline solution, pickled and passed through a series of electrolytic cells containing a tin bearing solution. Following the actual plating operation the strip must be rinsed before being processed further. Typically the rinsing takes place in three stages; the dragout recovery, the spray wash and the hot rinse. In the dragout recovery tank the strip is dipped into the rinsing solution (water contaminated with plating solution) and is further rinsed by a set of sprays followed by wringer or squeegee rolls. The solution which is sprayed onto and wiped off of the strip flows into the recovery tank. Solution not fed back into the main system is discharged to the sewer. This discharge is the most contaminated effluent from the halogen plating process and is made up of the same constituents as the plating solution but is in a form too dilute to reuse. The major pollutants in this stream are tin, cyanides, fluorides and suspended solids.

Several concepts for treating this primary dragout rinse had been proposed. These processes included ozonation and alkaline chlorination for destruction of cyanide wastes, calcium precipitation for the removal of fluorides, tin hydroxide formation and ion exchange for the removal of tin and thickening for removal of suspended solids. It appears that these processes may be workable; however, in most cases, the capital and operating costs for these systems are high and the space requirements for these processes preclude their installation in existing plating shops. Also, none of these processes is easily adaptable to the concept of zero waste discharge.

The use of countercurrent rinsing was suggested by personnel at National Steel Corporation as being a process which could achieve a zero discharge at minimal capital and operating expense as well as being a system which would not require excessive space. The process involves the use of countercurrent rinsing to concentrate the dragout wastes sufficiently so that they may be returned to the plating solution storage tank and reused. Computer simulations were run on the system and it was found that the number of countercurrent rinsing tanks required to achieve the necessary concentration in the first rinse tank was not excessive and could be retrofitted into an existing plating line. The main problem anticipated with the system was the buildup of sludge at an excessive rate in the plating solution. It was felt that this sludge buildup could be controlled by a chemical identified as AAAO. This chemical, developed by National Steel Corporation in conjunction with E.I. DuPont DeNemours and Company, Inc., minimizes the formation of this sludge in the plating process.

General Objectives

The general objective of this project was threefold:

1. To successfully design and to install a countercurrent rinsing system on an existing halogen tinplating line;

2. To demonstrate that over a long period of time the recycle of the primary dragout rinse could be achieved without loss of product quality or frequent solution changes;

3. To demonstrate that the system would significantly reduce the amount of pollutants discharged from a halogen tin plating process, perhaps even to achieve zero discharge.

V. CONCLUSIONS

A countercurrent rinse system was designed and installed on the Western Diesel Division No. 6 Electrolin line. A four stage system was designed for the recovery of the dragout electrolyte.

No design problems were encountered mechanically in the four stage rinse unit. Equivalent or improved rinsing of the strip resulted from the use of this new system. No problems with sludge buildup were encountered.

Problems resulted due to buildup of chlorides in the main electrolyte upon reuse of the concentrated rinse stream containing the recovered dragout. Methods to solve this problem are being investigated to enable reuse of the rinse water back to the main plating system. An alternate method for recovering the chemical values of the recovered dragout is also a definite possibility.

III. RECOMMENDATIONS

It is recommended that a second phase be instigated to evaluate both the recycle of the concentrated rinse into the main (on line) bath and indirect recycle involving the Detinning Plant.

The problems uncovered in the direct recycle of the concentrated rinse from countercurrent rinse system may be solved by a new technique in removing sodium ions from the rinse stream or the main bath. Another system was also investigated.

Plans are under way to recover immediately the tin values from the dragout recovery system by sending the rinse solution to the Detinning Plant. Full recovery of the fluorides and other constituents at the Detinning Plant is also under investigation. This method for recovery may be the most desirable after all the test results are evaluated.

IV. DESCRIPTION OF PLATING PROCESS

The Halogen Tin Process is designed to plate tin at high current densities and to produce high-quality tin deposits required for continuous electrotinning of strip steel. The process was introduced commercially in 1965.

The halogen tin bath, as the name implies, is formulated with halogen salts. The bath is operated with a relatively high chloride concentration to obtain maximum conductivity. Fluoride functions largely as a complexing ion in stabilizing the stannous tin. It combines with the tin to form a sodium fluorostannite complex salt, the composition of which may be expressed as Na_2SnF_4. In the absence of fluoride, stannous chloride hydrolyses in the pH range used to form precipitated hydroxy salts.

The halogen bath is subject to oxidation when operated under highly aerated conditions such as are encountered in high speed strip plating lines. The presence of small amounts of iron or copper salts in the halogen bath acts as an accelerator in promoting the oxidation. Without an inhibitor, substantial amounts of stannic tin are formed in the bath through oxidation. In addition to depleting the stannous tin, oxidation causes a significant fluoride loss from the electrolyte since each mole of stannic tin combines with six moles of fluoride to form sodium fluostannate (Na_2SnF_6). In contrast to the high solubility of the sodium fluostannite salt (Na_4SnF_6) the sodium fluostannate is relatively insoluble in the electrolyte and separates as a sludge. Addition agents are added to the bath in order to deposit the fine grain type of tin required for continuous electrotinning of strip steel. Without additives, the tin deposit is nonadherent and spongy.

With the high cost of tin and tin salts along with the costs of the fluorides and addition agents, each gallon of electrolyte represents over one dollar in value.

The halogen bath is used in a horizontal type process configuration (see Figure 1). This design comprises a succession of small horizontal cells. The plating solution is circulated continuously through each cell. Electrical contact is made to the strip by the rolls at each end of the cell. The tin anodes are positioned close to the strip travel path, which is just under the solution level, and rest on a center contact support and end support within each cell.

After passing through the plating cell, the strip is passed through a reclaim rinse (see Figure 2) in which the residual plating solution may be removed. The bright, lustrous finish of electrolytic tinplate is obtained by fusing and quenching the matte tin coating resulting from the plating process.

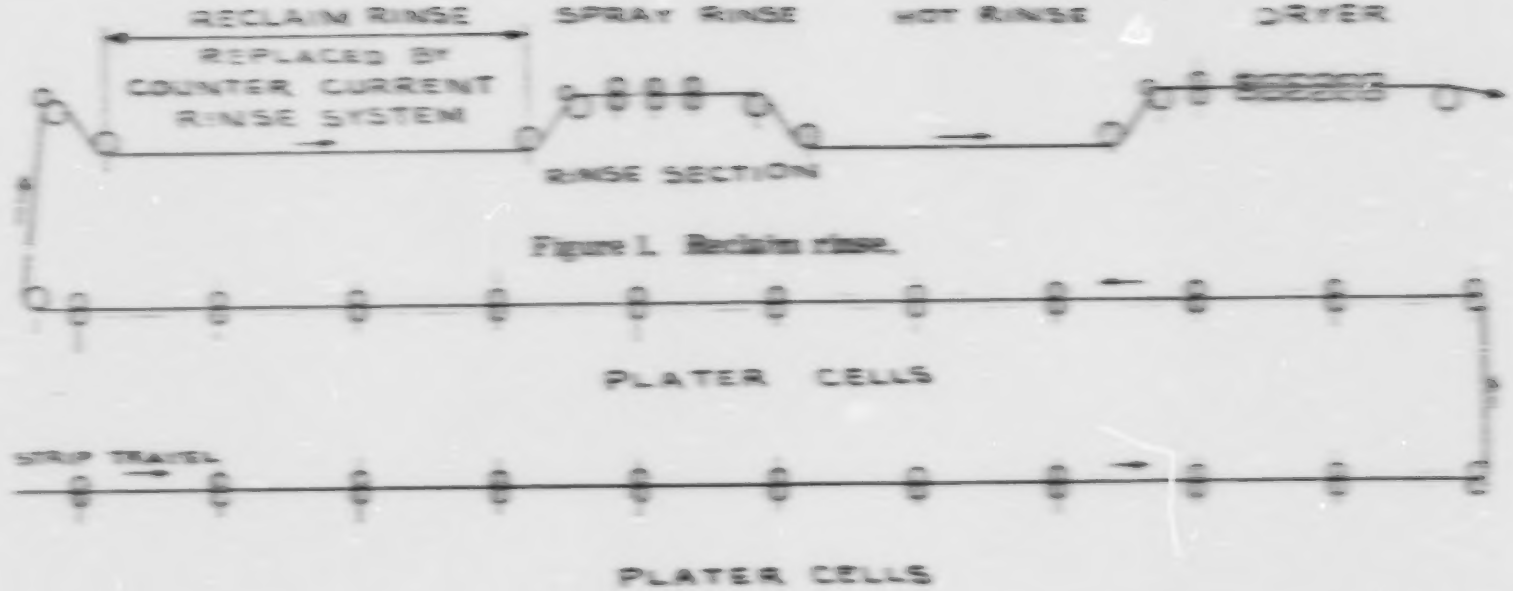

Figure 1. Reclaim rinse.

Figure 2. Horizontal pass acid electroclaning / alligeration (halogen process).

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V-THEORY AND DESCRIPTION OF COUNTERCURRENT RINSE PROCESS

In the production of tinplate, it is essential that after the strip leaves the last plating cell the electrolyte be rinsed completely from the surface of the tinplated strip. It has been the practice in the mill to employ ample quantities of rinse water to effect this removal and to insure the cleanliness of the final product. However, with this practice only a small amount of the electrolyte removed from the last plating cell on the surface of the strip can be recovered and reused in the plating system. Since water that is put into the plating system is limited to only the amount needed to maintain volume, the bulk of the rinse water goes into the sewer and is a contributor to stream pollution. In order to effect a higher percentage of recovery of dragout, the volume of rinse water must be decreased and the concentration of electrolyte increased. A method that can do this is countercurrent rinsing.

As the name implies, the flow of rinse water is counter to the flow of the strip. The main advantage of this type of rinsing is the ability of a much smaller quantity of water to effect the same degree of dragout removal from the strip. This insures the same cleanliness of the strip after rinsing and results in a smaller volume of rinse water that can all be used as makeup back in the main system. This volume has a high concentration of electrolyte salts and effects almost total recovery of dragout.

ADAPTATION TO MILL USE

Figure 2 shows the outline of a typical reclaim rinse tank into which the strip passes immediately after the last plating cell. The dotted line shows the present path of the strip through the reclaim tank. The strip is deflected down into the rinse water around sink roll (J_O) from the wringer rolls (F_O and E_O). The strip emerges from the tank from sink roll "J_1" into wringers "I_1" and "I_2." From this set of wringer rolls, the strip is further washed in the hot rinse tank.

To employ the principles of count rourrent rinsing, the tank now in place, or a new tank to replace the existing tank, must be divided into compartments. A four-stage system is shown in Figure 3. The flow of rinse water must be opposite to the direction of strip travel. The flow proceeds through each compartment by the proper positioning of overflow weirs. The level of rinse water is lower in each succeeding compartment or stage. The same rinse water flows through each stage. There are two ways in which the strip may be contacted with the water in each compartment. Sink rolls could be placed below the solution level in each compartment. The strip would thus be diverted down into each compartment and would exit through wringer rolls located between compartments. The second method, would be to pass the strip directly across the top of the compartments as shown in the sketch. The solution in each compartment would be sprayed onto the strip from pumps as indicated. The solution would drain back into each individual compartment at the entrances of the wringer rolls, such as (F_O-F_1), (G_O-G_1), (H_O-H_1) and (I_O-I_1). In the opinion of the operating

people; this method would have advantages. All sink rolls would be eliminated. These rolls are sources of trouble since their positions make them difficult to clean. Also strip breaks at these locations would entail more down time.

It is believed that with the proper size pumps and good spraying practice, the mixing of solution in each stage with the drag through films can be as good, or better, than with the design involving sink rolls. One control is required on the feed water at point A. When the line is not running this valve must be closed to prevent dilution of the system. The flow of the rinse water is A to B to C to D to E.

With a three or four stage system the mathematics indicate the possibility of recovering over 90 percent of the dragout electrolyte. Good mixing of the solution in each stage with the film on the strip is required in approaching the theoretical performance. Cost savings should be considerable especially when all the electrolyte lines are considered on a yearly basis.

Using the math model shown in Figure 3, the equilibrium concentrations in each rinse tank of a countercurrent system having "N" tanks can be found by solving "N" simultaneous material balance equations. One equation is written for each tank. A typical equation for the n^{th} tank is:

$$DX_{n-1} - (D + R)\, X_n + R X_{n+1} = 0$$

where X_n = the equilibrium concentration in the nth rinse tank = ounces/gal
D = dragout flow rate = GPM
R = countercurrent flow rate = GPM
$X_0 = X_n$ = the cell concentration (a constant) ounces/gal

If $K = \frac{R}{D}$, the countercurrent flow ratio, is substituted, the typical n^{th} equation becomes:

$$X_{n-1} - (1 + K)\, X_n + K X_{n+1} = 0$$

After solving sets of these equations for 2, 3 and 4 tank systems, it was found that the solution for the equilibrium concentration in the nth tank of a system of "N" tanks could be generalized as follows:

$$X_n = \frac{\sum_{i=N-n}^{i=0} (Ki)}{\sum_{i=N}^{i=0} (Ki)} X_0$$

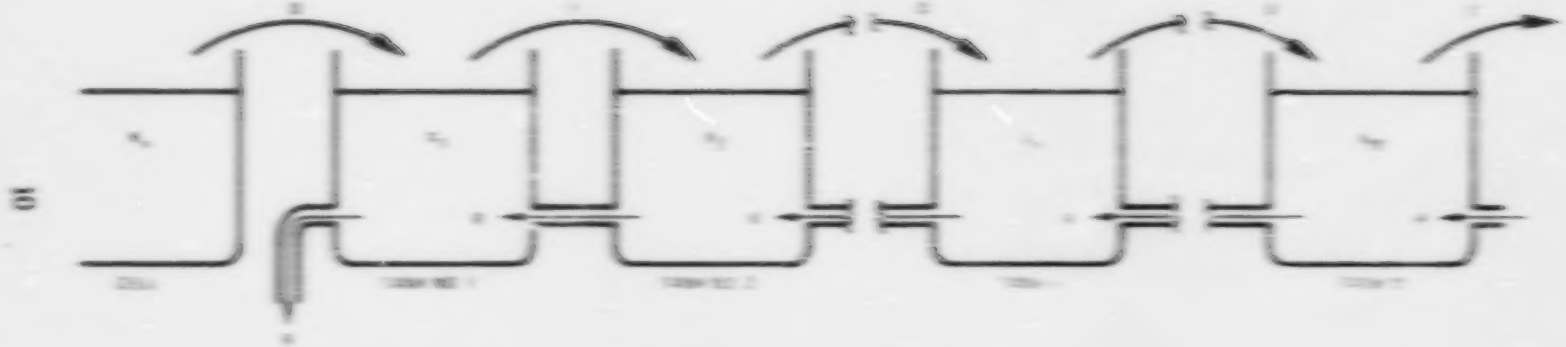

Figure 2. Math model.

As an example, the equilibrium concentration of the 2nd tank in a 4 tank system with a countercurrent flow ratio of 2, may be found by setting n = 2, N = 4, and γ = 2.

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$$X_2 = \frac{\sum_{i=0}^{2} (2^i)}{\sum_{i=0}^{4} (2^i)} X_0$$

$$X_2 = \frac{2^0 + 2^1 + 2^2}{2^0 + 2^1 + 2^2 + 2^3 + 2^4} X_0$$

$$X_2 = \frac{1 + 2 + 4}{1 + 2 + 4 + 8 + 16} X_0 = \frac{7}{31} X_0$$

$$X_2 = .2258 X_0$$

The results for 2, 3 and 4 tank systems are given in Figures 4 to 6. As an example, in a two stage system the concentrations of the rinse water in either tank can be found from the graph in Figure 5. If the ratio of water flow to dragout from the last plating cell into the rinse system is two, the equilibrium concentration in stage #1 is 66 percent of the concentration of the electrolyte in the cells. The equilibrium concentration in the second or last stage is 16 percent of the bath composition. If the ratio of rinse water to dragout increases the equilibrium compositions will decrease according to the graph.

A four stage system was chosen for this installation for several reasons. Space was a primary consideration. The graph for a four stage system, Figure 6, indicated low concentrations could be obtained for the final rinse and high concentrations in the first rinse. Thus product quality could be maintained or improved plus a concentrated stream would be generated for closed loop recycling or use in a recovery system. A five stage system would be harder to maintain and no big advantage over a four stage system. A three stage system would not give enough difference between the first and last tanks.

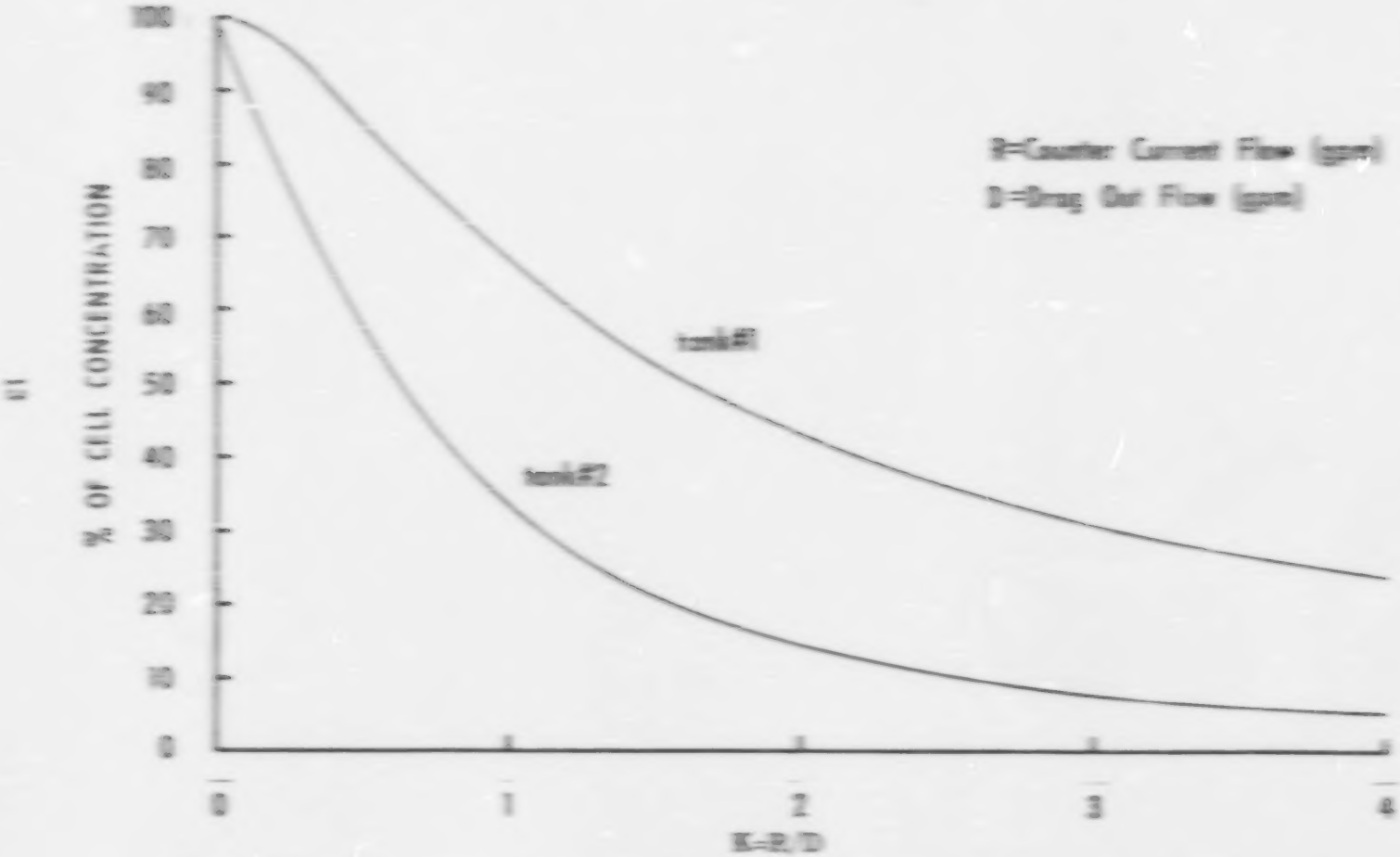

Figure 4. Rinse tank equilibrium concentration as percent of cell concentration (two tank system).

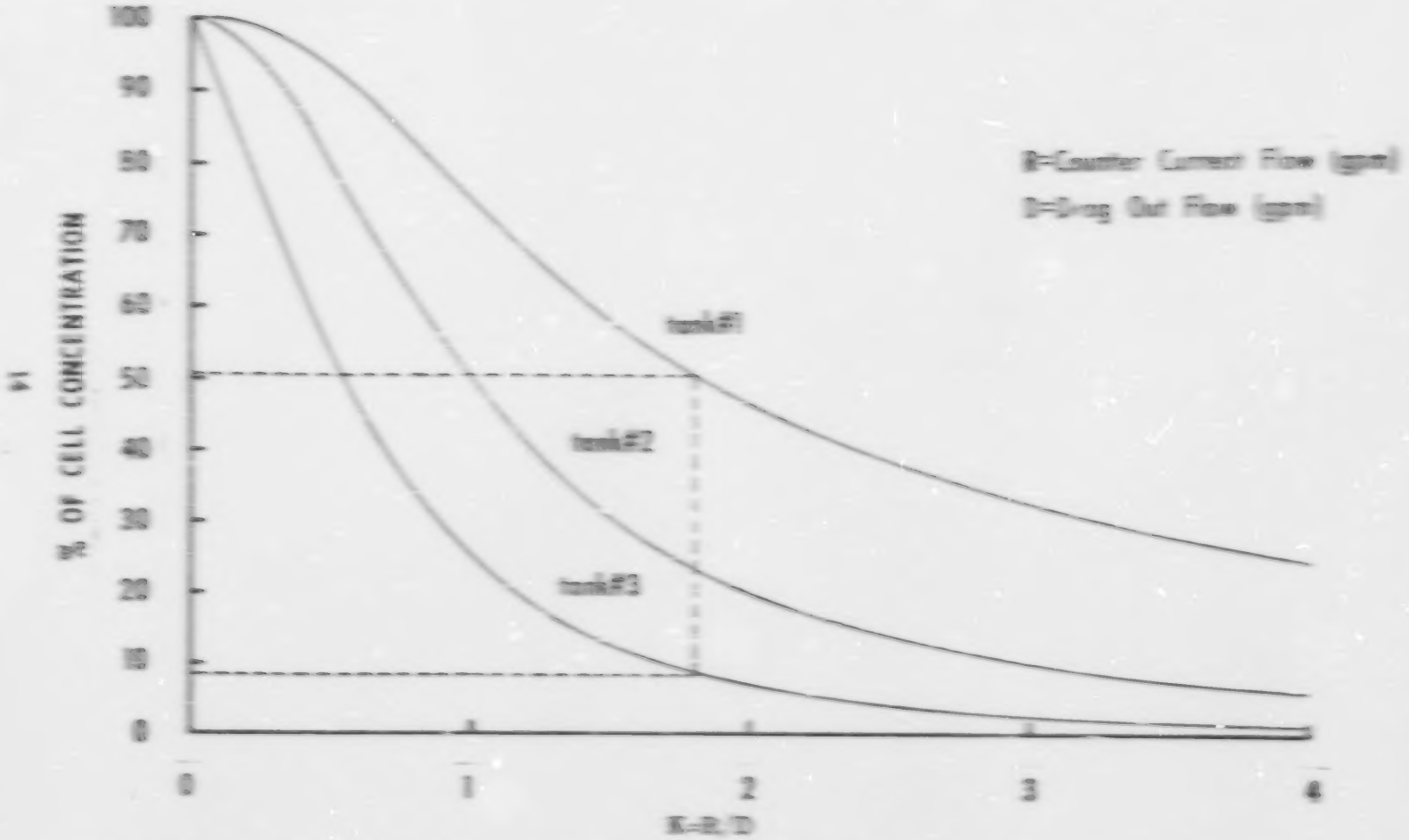

Figure 5. Rinse tank equilibrium concentrations as percent of plating cell concentration (three tank system).

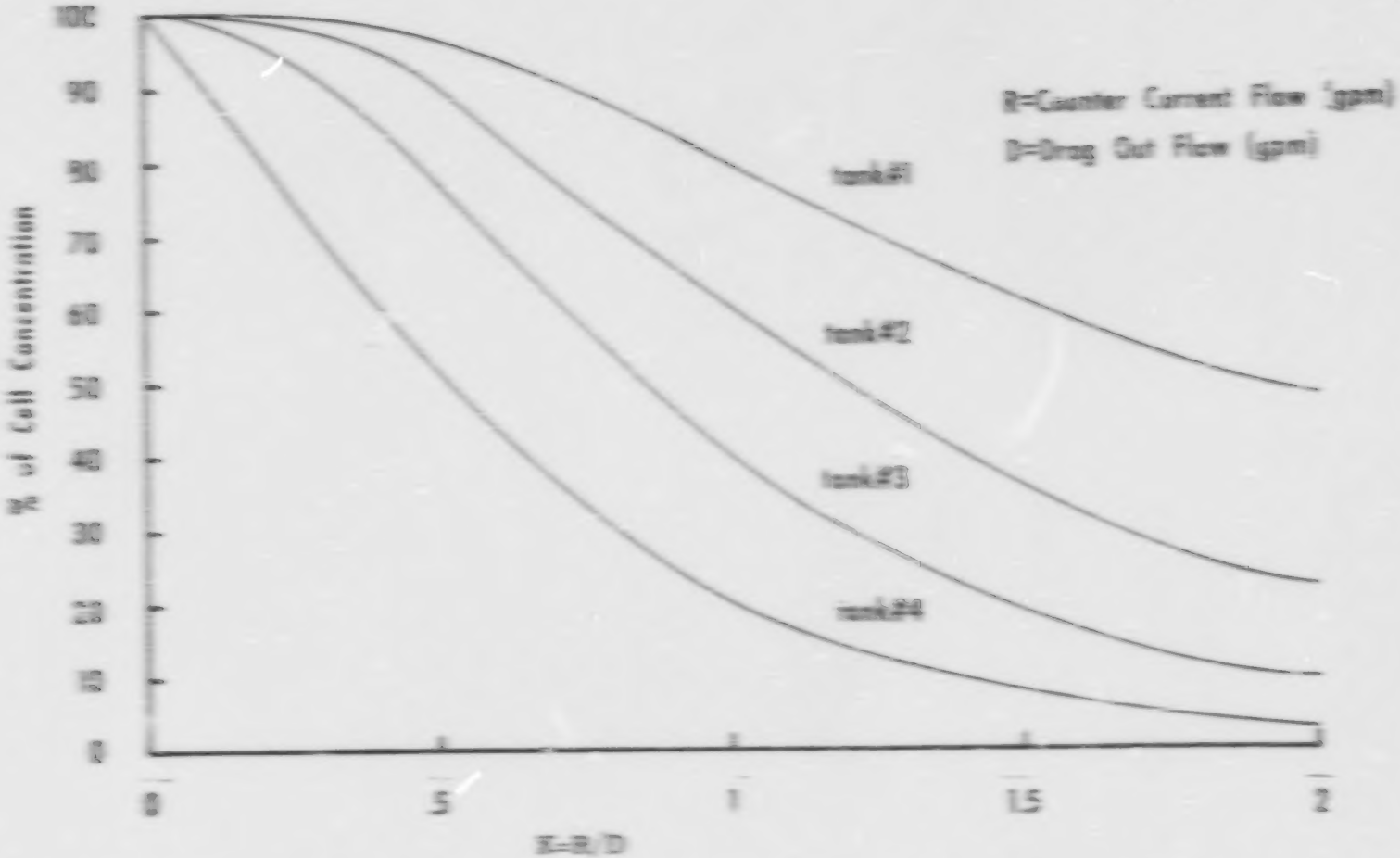

Figure 6. Rinse tank equilibrium concentrations as a percentage of cell concentration. Four tank system.

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VI-DESIGN AND INSTALLATION OF COUNTERCURRENT RINSE EQUIPMENT

The No. 6 Electrolytic Tinning Line was designed in 1964. The line was built to produce tin plate coils as follows:

LINE SPECIFICATIONS	
Maximum Coil Weight	60,000 lb.
Minimum Strip Thickness	0.003 in.
Maximum Strip Thickness	0.036 in.
Minimum Strip Width	16 in.
Maximum Strip Width	45 in.

LINE SPEED	
Entry End	1500 fpm at 240 volts
	2400 fpm at over voltage
Center Section	600 fpm at 240 volts
	2000 fpm at over voltage
Exit End	1000 fpm at 240 volts
	2400 fpm at over voltage

The line is 1,250'-6" long. The plater cells are located in the center section. The reclaim tank is located immediately after the last plater cell on the third deck of the plater structure. The original tank was equipped with an entry and and exit dunk roll to submerge the strip for 25'-6" in the reclaim water rinse.

Specifications were drafted for the replacement of the existing dunk-type reclaim tank section with a cascade, wringer roll type rinse tank. The scope of work included removal of the reclaim tank, modifying the adjacent tanks, alterations to the structural supports and installation of the new equipment.

The new reclaim rinse tank has four compartments for circulating rinse solution. Fresh demineralized water is added to the No. 4 or last compartment. Each compartment overflows in a countercurrent direction from compartment No. 4 to compartment No. 1. The individual compartments have a pump suction line for the spray pumps. The No. 1 compartment is furnished with an overflow line.

The spray pumps take solution from the individual tank compartments and pump to the spray headers in the same tank compartment. Each compartment is also furnished with a hopper for addition of chemicals.

NEW EQUIPMENT SPECIFICATIONS:

Tank	= Fabricated of 5/8" thick mild steel, 1/4" rubber lined and 3/16" rubber covered.
Cover	= Fabricated 1/4" thick mild, steel 1/4" rubber lined.
Drive Bases	= Fabricated 5/8" thick mild steel.
Pump Bases	= Fabricated 5/8" thick mild steel.
Rolls	= Nine (9)=10" diameter and 54" face steel, rubber covered. One (1)=15=1/8" diameter by 54" face steel, rubber covered.
Roll Drives	= Ten (10)=single reduction gear type with steel housing.
Roll Drive Motors	= Nine (9)=3 Horsepower, 1150/2300 rpm One (1)=5 Horsepower, 1150/2300 rpm
Couplings	= Gear Type
Bearings	= Pillow Block Roller Bearing Type
Pumps	= Four (4)=100 gpm at 50 psi
Pump Motors	= Four (4)=15 Horsepower, 1750 rpm
Spray Headers	= Eight (8)=316L Stainless Steel

The tank, drive bases, and pump bases were mounted to a common drip pan. The rinse tank being on the third level of the structure, the drip pan was installed to prevent any spillage from dropping on the structure and/or equipment on the two levels below. The drip pan also served as a unitized sub-base for the equipment. After all equipment was mounted to the pan it was then possible to fabricate and install all piping and apparatus at an off-site location.

The plating line operation was shut down for four (4) days to install the countercurrent rinse tank. The installation was possible in such a short time because of the pre-assembly of equipment and piping. The old reclaim tank was removed, and the new rinse section dropped into place in two (2) lifts of an overhead crane. The balance of the installation time was used to join the new tank section to the existing equipment and connect the piping and electrical terminations.

A schematic of the piping system is shown in Figure 7. The incoming water flows through a rotameter, rated 0 to 5 gpm. This water flows in compartment No. 4. The overflow from compartment No. 1 flows through a rotometer on the way to the main plating system.

The value on the incoming water line is controlled by a solenoid which closes the valve whenever the speed of the line reaches zero. This protects against dilution of the equilibrium concentrations during shut down periods.

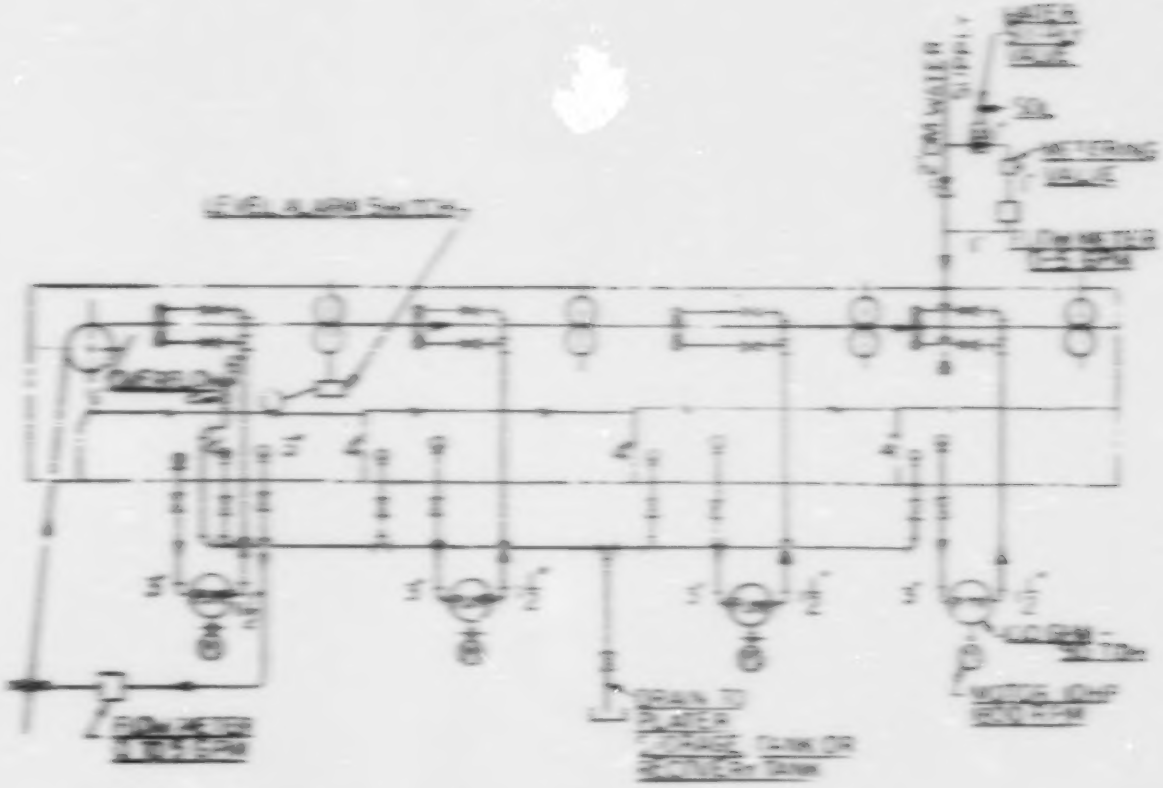

Figure 1. Multistage spray rinse tank piping system.

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VII-OPERATIONAL RESULTS

A. OPERATING PRACTICES AND EXPERIENCE

Production with this new unit began on November 26, 1974. Problems that were anticipated during start-up such as buildup on the rolls and drying out of salts on the strip did not occur.

Sodium bifluoride was added in the No. 4 compartment. This chemical maintains an acid rinse so that the tin in the solution does not hydrolyze and stain the strip. It was found that one pound per hour maintains a pH of 3.5 to 3.8 which is about the same as the plating solution.

After the initial week, the spray nozzles were found to be corroding. These were replaced with stainless steel nozzles and no major problems have occurred with the spray nozzles as long as they were cleaned on a weekly basis.

The rolls need only occasional sanding to remove salt buildup. The solution of the No. 1 compartment has a pH of 3.5 to 3.8 and the total tin concentration ranges from 0.5 to 1.0 ounces per gallon. The No. 4 compartment ranges from 0.10 to 0.15 ounces per gallon. The demineralized water is put into the No. 4 compartment at a rate of 3 to 3.5 gallons per minute.

On start-up of the countercurrent rinse tank, it was found that the quality of the tinplate was as good as, or better than, before. The tinplate strip is brighter because the countercurrent rinse tank affords better rinsing of the strip. It is only natural that a cleaner water is used to rinse the strip with the countercurrent rinse system versus the old method of immersion rinsing. Since the strip is rinsed better before it goes into the scrubber and the hot rinse tank, there are less salts left on the strip which allow for a brighter strip when tin is melted in the reflow.

Eight sets of spray headers are used, two sets in each tank. Each header has eight stainless steel V-jet sprays. The sprays are located five inches apart.

8. RINSING PERFORMANCE

In Table 1 the results of the chemical analyses for stannous tin are given. It required three days or 72 hours to reach equilibrium concentrations. Calculations had indicated at least 48 hours would be required. Once equilibrium was reached, the differences in concentrations of the four stages were as expected.

Because of the high flow required to maintain volume in the main plating system, the concentrations in the various stages have not reached the higher levels expected. This, however, is a big advantage because the concentration of the last stage (No. 4) can be kept at a very low level. This means minimum loss of chemicals to the spray and hot rinse sections. It was somewhat surprising to find that a flow rate of 4 to 5 gpm into the countercurrent rinse could be used without a buildup of volume in the main plating system. Apparently the evaporation losses are considerable.

The concentration of the tin in the last stage is about one tenth of the concentration found in the reclaim tank during the base period investigation. Therefore losses to the spray rinse and hot rinse sections have been reduced by around 90 percent. This figure will be checked in future repeat testing by Environmental Control of the effluents from these rinses. This reduction is in addition to the elimination of the losses which occurred from the flow which used to enter the sewer from the reclaim tank.

Sodium bifluoride is being added to the last stage of the countercurrent rinse system to control pH and prevent hydrolysis of tin salts on the strip. Operators have noted that the strip looks cleaner than it did when the old system was in use.

Table 1. Concentrations of Stannous Tin in Each Stage

	STAGE(gm/gal (SN + TIN)*)			
Date	No. 1	No. 2	No. 3	No. 4
11-26-74	0.18	0.09	0.06	*
11-27-74	0.18	0.09	0.06	*
11-28-74	0.37	0.22	0.12	0.03
11-29-74	0.49	0.30	0.12	0.06
11-30-74	0.40	0.21	0.09	0.06
12-2-74	0.43	0.23	0.09	0.03
12-4-74	0.49	0.34	0.18	0.06
12-6-74	0.49	0.15	0.12	0.06
12-9-74	0.31	0.15	0.09	0.03
12-11-74	0.59	0.31	0.15	0.09
12-13-74	0.46	0.21	0.12	0.06
12-20-74	0.43	0.21	0.12	0.06
12-27-74	0.46	0.24	0.12	.06
1-31-75	0.69	0.34	0.15	0.09
2-28-75	1.08	0.55	0.27	0.15
3-14-75	0.65	0.30	0.15	0.09
4-10-75	0.62	0.31	0.15	0.06
5-8-75	0.92	0.33	0.12	.04
5-19-75	0.86	0.37	0.15	.05
5-25-75	0.68	0.31	0.12	0.06

* Values should be multiplied by factor 1.2 for total tin.

C. FIRST PROBLEM OF CHLORIDE BUILDUP

Throughout December of 1974 a rise in the chloride content of the electrolyte was noted. A plot of the chloride content is shown in Figure 8. As can be seen by the graph the chloride level prior to the start-up of the countercurrent rinse system was between 4.0 and 4.6 ounces per gallon. After the start-up of the new rinse system on November 26, the chloride content started to rise. A level of 5 ounces per gallon was reached on December 13; a level of 6 ounces per gallon was reached on December 22 and finally a level of 7 ounces per gallon was reached shortly after the first of the new year.

The levels of tin and fluoride did not increase. The reasons for the increase in chloride have become obvious. Chemical additions were reduced for several components which normally supply hydrogen ions to the plating bath. Both stannous chloride and sodium bifluoride are acid salts. With a cut back in the additions of these chemicals more hydrochloric acid was added to control pH.

The desired pH range is 3.3 to 3.5. The pH will gradually rise due to the electrode reactions at the cathode (strip) and also due to the formation of sludge. According to the DuPont Manual, one of the major causes for change in pH during operations is the difference in anode and cathode efficiency. Hydrogen ions are consumed in the formation of hydrogen at the cathode. Another cause for increase in pH according to the DuPont Manual is the oxidation reaction. This can be expressed as follows:

$$2Sn^{+2} + O_2 + 4H^+ \rightarrow 2Sn^{+4} \text{ and } 2H_2O$$

Since the reaction removes hydrogen ions, the pH increases.

To counteract this rise in pH, hydrochloric acid was added. During the rise of chlorides in December, it was hoped that an equilibrium concentration would be reached before salting out occurred. This is a condition which occurs when the total salt content rises too high and crystallization develops in the plating system, especially on rolls, causing denting of the surface of the strip. When the chloride content exceeded 7.0 oz/gal, the dent problem developed and corrective action had to be taken. Steps were taken which included running part of the effluent from the countercurrent rinsing system to the sewer rather than back into the plating system.

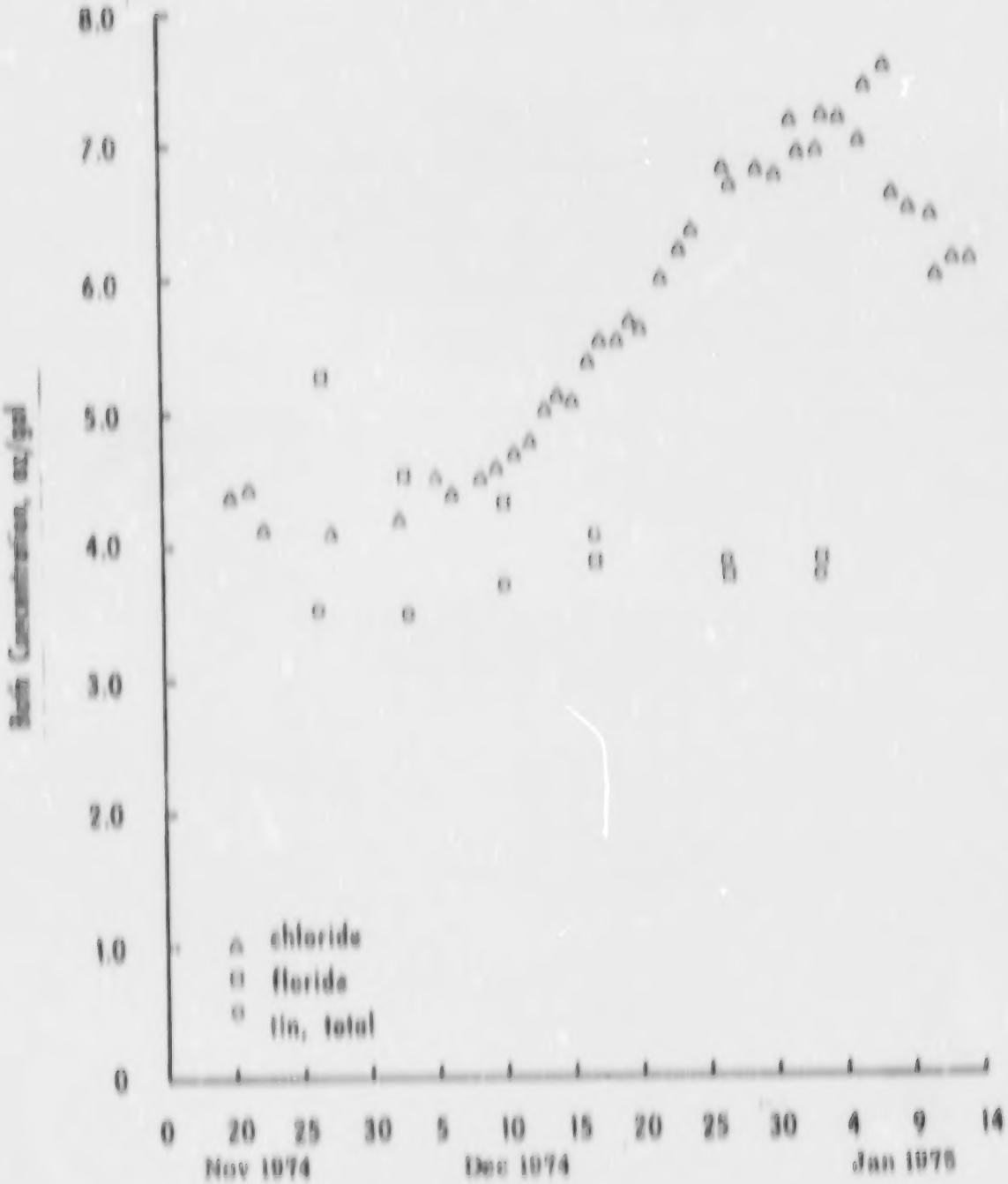

Figure 8. Bath concentration vs. time.

D. ECONOMIC STUDY FOR ONE YEAR OF OPERATION

In Figure 9 a graph shows the consumption of stannous chloride and hydrochloric acid for the year (1974) prior to the installation of countercurrent rinsing and the first year of its use (1975). The amounts are given as the chlorides of each of the two chemicals. Taking an average for each twelve month period, see Figure 8, the amounts used per month were -

	1974	1975
$SnCl_2$ (Cl)	6,000 lbs	3,000 lbs.
HCl (Cl)	1,000 lbs	2,200 lbs

As the consumption of stannous chloride was reduced the use of hydrochloric acid had to be increased to obtain the acidity required to maintain the pH within the desired range (3.4 to 3.7).

The line was operated at a slightly higher pH in order to control the level of chlorides below a level where salting out would occur. This will be discussed in more detail later. However, with certain slight changes in operation the tin plating line was able to operate without denting problems at least until November 1975.

The 3,000 pound difference in chlorides from the reduction in the rate of consumption of stannous chloride is equivalent to $\frac{3000}{0.63}$ or 4,762 pounds per month of stannous chloride. Taking a cost of stannous chloride at $2.60 per pound this represented a savings of $12,380 per month. From this must be subtracted the increased cost in the use of hydrochloric acid. The 1,200 pounds per month increase amounted to $60 per month based on a cost of $12 per ton of 32% acid. The net savings was thus $12,380 - $60 or $12,320 per month or $147,840 per year. There were other changes made during the year by Operations which may have assisted in this cost reduction. Additions were made more often which insured more complete dissolution of the chemicals. This change was made in the summer of 1975.

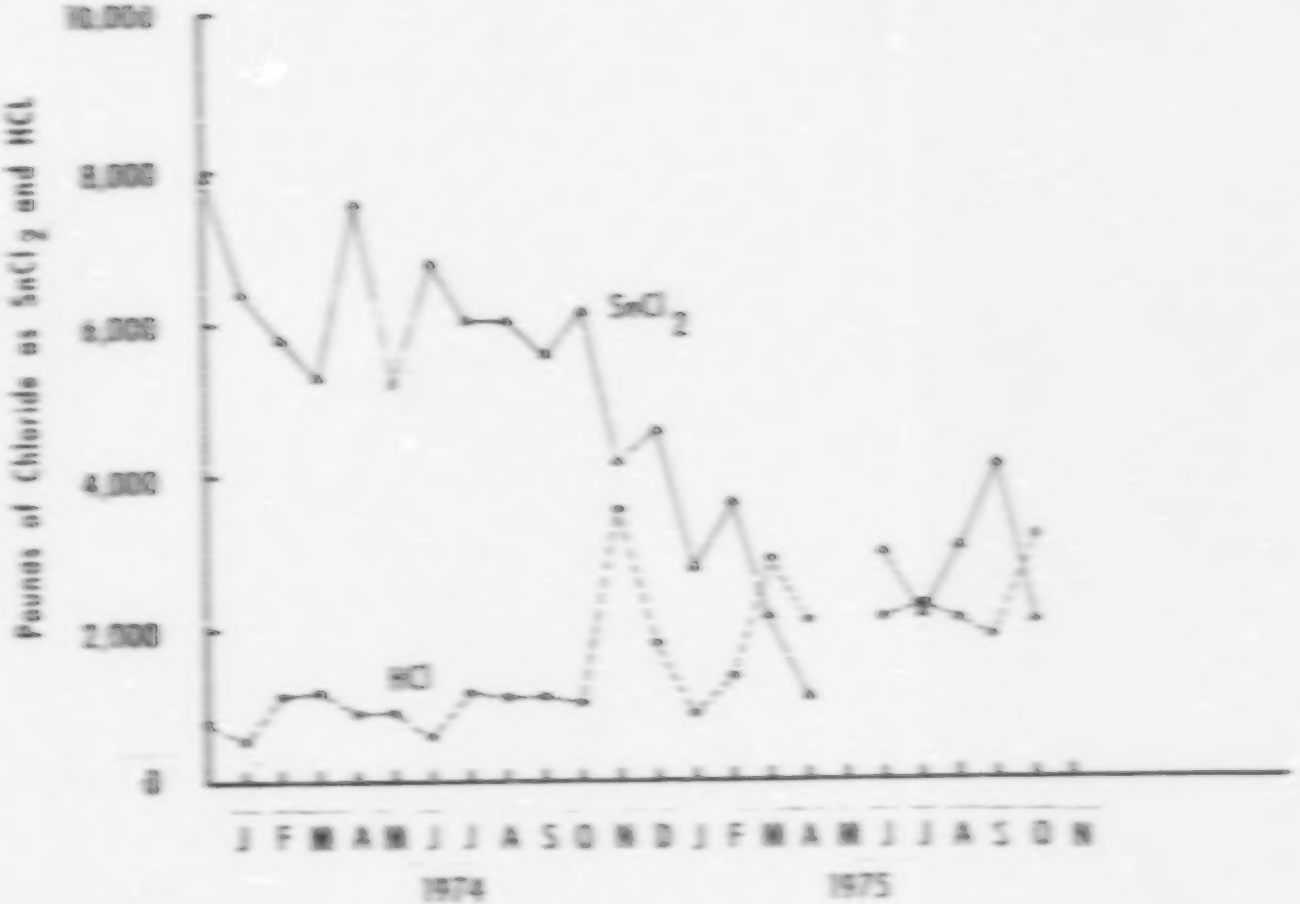

Figure 1. -Rate of monthly consumption—No. 6 electro-line.

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8. PROBLEMS GENERATED

After one year of operation an evaluation has been made of the new rinse system. Some problems have developed not due to the mechanical performance of the equipment but due to the peculiarities of the Halogen Tin Plating Process. It was anticipated prior to the installation that this might be the case and some modification of the process or operation would be required to obtain maximum benefit from dragout recovery.

Since oxidation of tin in the halogen electrolyte occurs continuously, sludge is produced also continuously. Sludge contains sodium, tin and fluoride but very little chloride. There is, therefore, a continuous need for additions of sodium, fluoride and tin to replace the amounts lost in sludge formation. While part of the tin may be supplied by the differences in electrode efficiencies the other elements must be added by periodic chemical additions.

The electrolyte also becomes more basic with coulombic input due to the generation of hydroxide ions at the cathode and with the consumption of hydrogen ions in sludge formation. Therefore, regular additions of either stannous chloride or hydrochloric acid must be made for control of the pH at a fixed value.

Another peculiarity of the halogen electrolyte is the need to keep the sodium chloride content below the level where salting out, or saturation occurs. Sodium chloride formation within the bath will lead to denting of the strip due to buildup on rolls. The anions, particularly fluorides and chlorides, must be held within a certain range to avoid this problem.

In Figure 10, the amount of chlorides in the system is shown. After the initial surge of buildup in January the levels were reduced by operating the electrolyte at a slightly high pH thus reducing the additions of hydrochloric acid. Operating personnel have contended that the denting problem has been greater than normal even during the first periods. In December 1979, the denting problem became severe. No time was given to countercurrent rinsing and the flow was diverted to the sewer from the first stage.

It has been found that the use of countercurrent rinsing along with dragout recovery and reuse has affected to some extent two aspects of the halogen process: (1) the control of pH and (2) the level of chlorides.

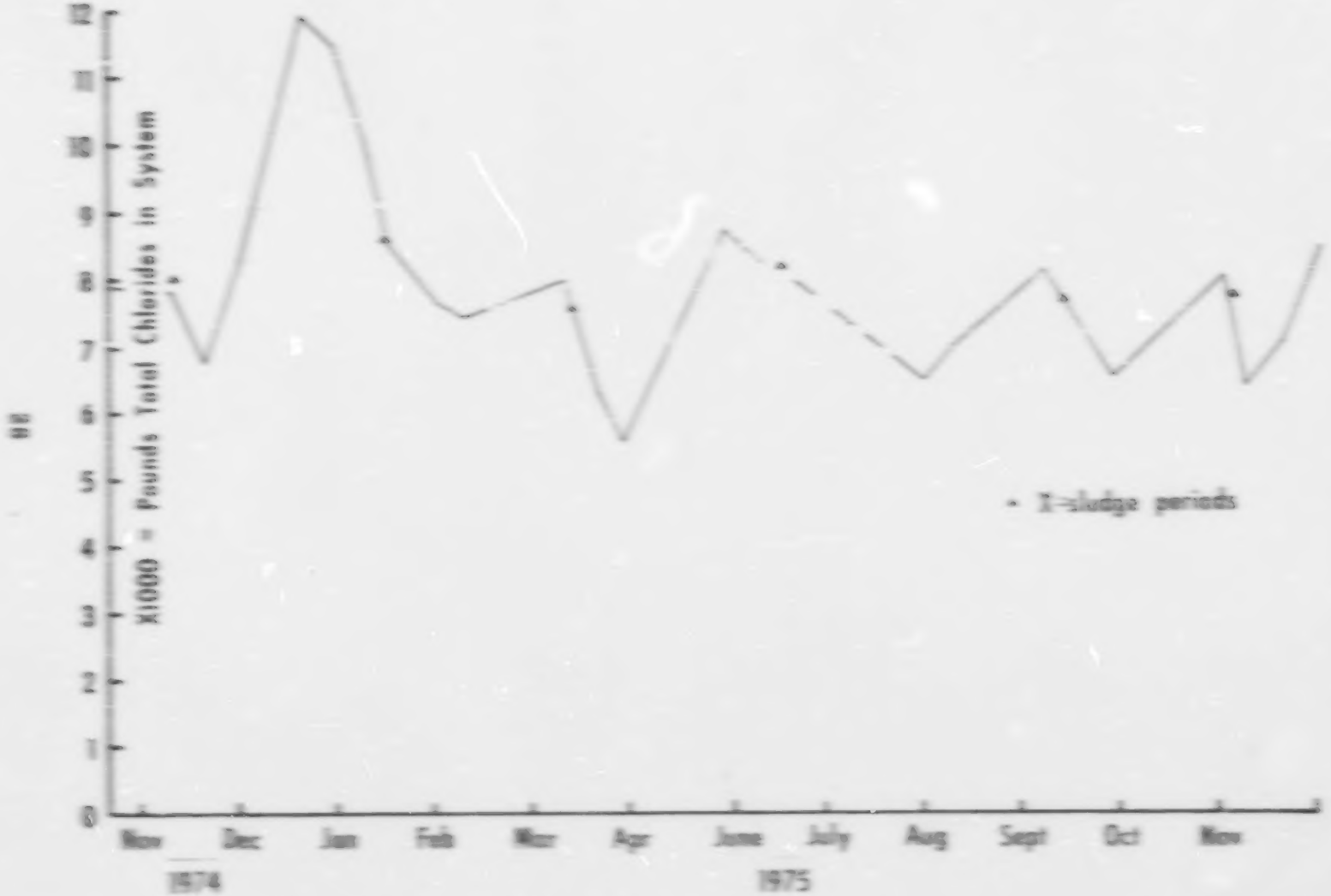


Figure 10. Amount of chlorides in system.

VIII-SOLUTIONS TO PROBLEMS GENERATED

A. USE OF HYDROFLUORIC ACID

As previously mentioned the problem of chloride buildup resulted because additions of hydrochloric acid were still required for pH control after recovery of dragout was instituted. Since chlorides are not removed from the system in the formation of sludge, the elimination of losses due to dragout results in an increasing concentration of chlorides in the electrolyte. A solution to this problem, as suggested by Dr. D. A. Swalheim of DuPont, is to substitute hydrofluoric acid (HF) in place of hydrochloric. Fluorides are removed from the system in sludge formation.

This possibility has been studied and is still under consideration. However, the problem with using hydrofluoric acid is the hazardous nature of this chemical. Burns due to hydrofluoric acid are extremely serious. Nevertheless this acid is used in many plants where proper safety precautions are employed.

In efforts to counter the reluctance of operating personnel to consider the use of hydrofluoric acid, the Environmental Department of R & D has designed a system for minimizing the hazards involved in unloading, transporting, and emptying containers of HF. This system consists of a portable heavy steel tank with a double valve control. A sketch of this system is shown in Figure 11. All the employee in charge of adding the HF has to do is open the air vent and turn the long stemmed valve on or off. When the container is empty, as indicated by a floating ball type, liquid level instrument, the nozzle is rinsed out via a special water connection and removed.

Another method for handling HF is shown in Figure 12 . This method appeared in the January 17, 1977 issue of Chemical Engineering. Air pressure is used to fill and transport to the user tank a predetermined amount of the acid. No pumps or carboys are required. With the storage tank located outside the building, the time that the acid is in the plant at full strength is limited to just a few minutes.

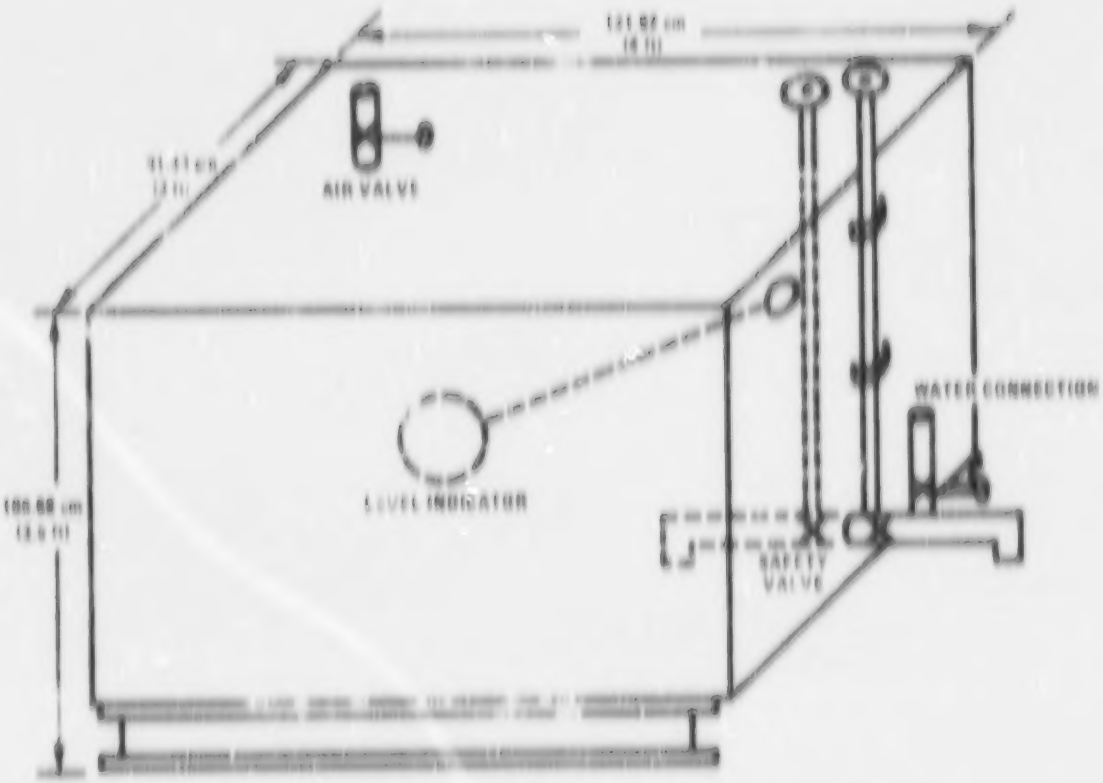


Figure 11. Proposed HF tank.

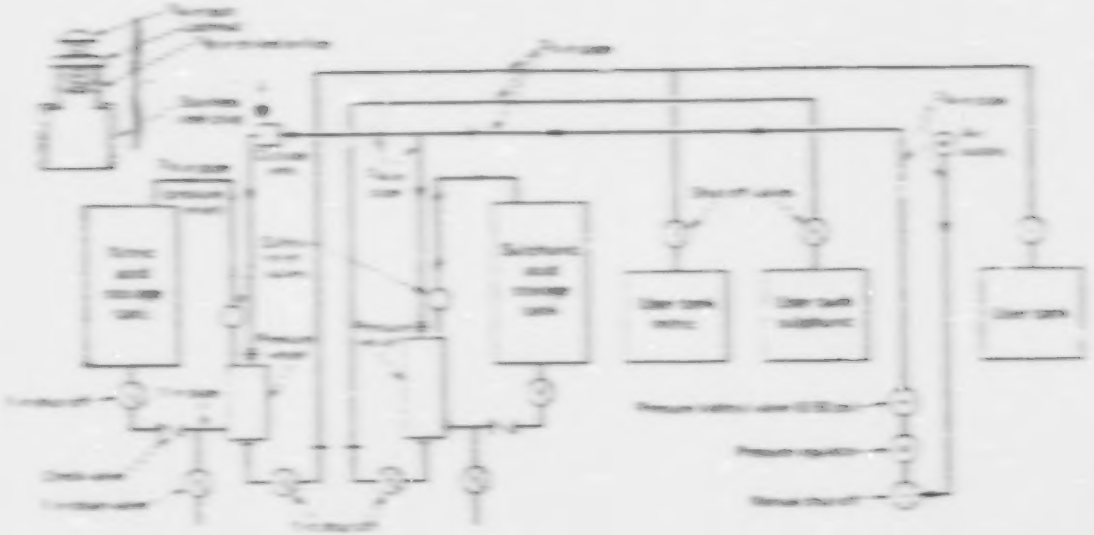
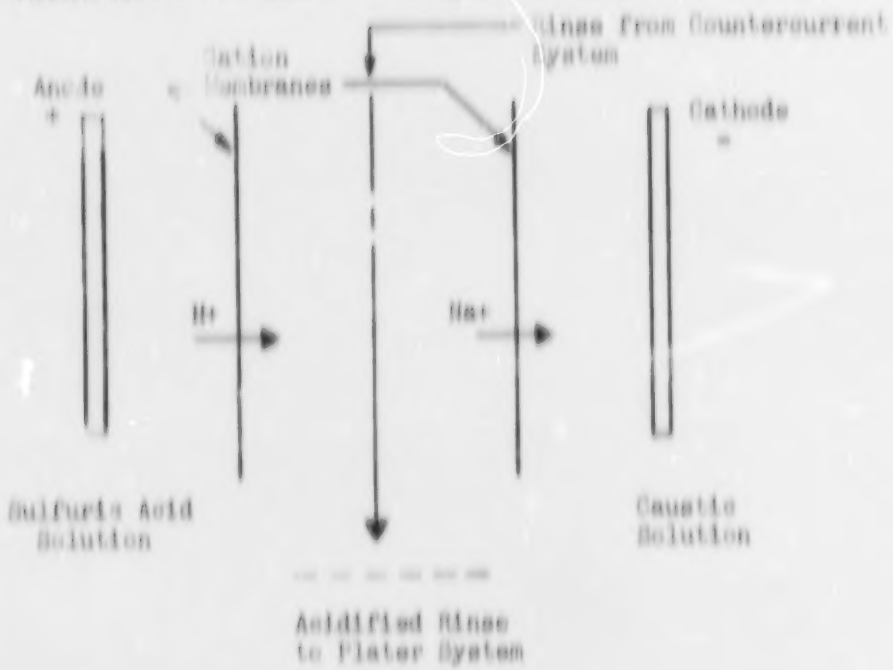


Figure 12. Distribution system for strong acid using neither pumps nor carboy. (© Chemical Engineering, 1/17/77, page 58; used with permission.)

B. USE OF AN ALKALINITY REMOVAL UNIT

Recent advances in technology has extended the use of electrodialysis cells from laboratory curiosities to commercial use. These cells employ permselective membranes, which have ion exchange properties. New fields for use of these cells include continuous ion exchange. In this process, one ion is exchanged for another, in the same manner as an ion exchanger, with the exception that in this process the resin (the membrane) is regenerated continuously by electrical energy, rather than cyclical, by chemical regeneration.

In the halogen bath there is a continued need to add acid to counter the buildup of sodium ions due to the oxidation of the tin and to the generation of hydroxyl ions at the cathode. As a result sodium hydroxide is produced. The concentrated rinse from the countercurrent rinse system can be made to pass between two cationic membranes wherein the sodium is migrated from the bath through the cation membrane to the cathode compartment forming sodium hydroxide. At the same time hydrogen is freed from the acid in the anode compartment into the concentrated rinse water. A diagram of the process follows:



Alkalinity is removed from the stream and acid is generated thus reducing or eliminating the acid requirements. In this manner, an alkalinity removal unit might permit operation of a closed loop system. If more removal of alkalinity is required to balance the system, electrolyte from the plating cells could be passed through the unit.

The only by-product from the unit is a solution of caustic soda. This material has wide usage for acid neutralisation. The purity should be sufficient to permit use in processes requiring solutions of caustic soda.

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C. <u>LIQUID-TO-RECYCLE VIA DETINNING PLANT</u>

Located at the Weirton Steel Division is a Detinning Plant, which was designed primarily for the recovery of tin from scrap tinplate. However, this plant also processes sludge from the plating lines. A flow sheet of the process is shown in Figure 12A. The sludge enters the system at the point where sodium stannate filter cake is fed to the dissolving tank. When sludge is being processed the sludge replaces the filter cake in the system. The concentrated rinse from the counter-current rinse system is now being sent to this plant for recovery of the tin values. The rinse solution replaces part of the water used to dissolve the sludge.

The chemical principles on which the entire operation is based are generally well known. Metallic tin will dissolve in a hot solution of sodium hydroxide to form soluble hydrated sodium stannate. Once the alkaline solution has become saturated with sodium stannate, precipitation occurs and the crystalline solid is periodically removed and separated from the detinning solution. It is then dissolved in water in a suitable tank. This is the same tank or point in the process where sludge or concentrated rinse solution is added. The stannate solution is transferred to a second tank where it is converted to stannic hydroxide by controlled addition of sulfuric acid. The tin hydroxide thus formed appears as a white insoluble compound. Sodium sulfate, the other product of the reaction, remains dissolved in solution as well as the fluorides from the sludge and recovered rinse. The tin hydroxide is next filtered and washed. The wet filter cake is then dried and mixed with anthracite coal. When this mixture of hydrated tin oxide and carbon is heated to about 2200°F, the oxide is reduced to metallic tin.

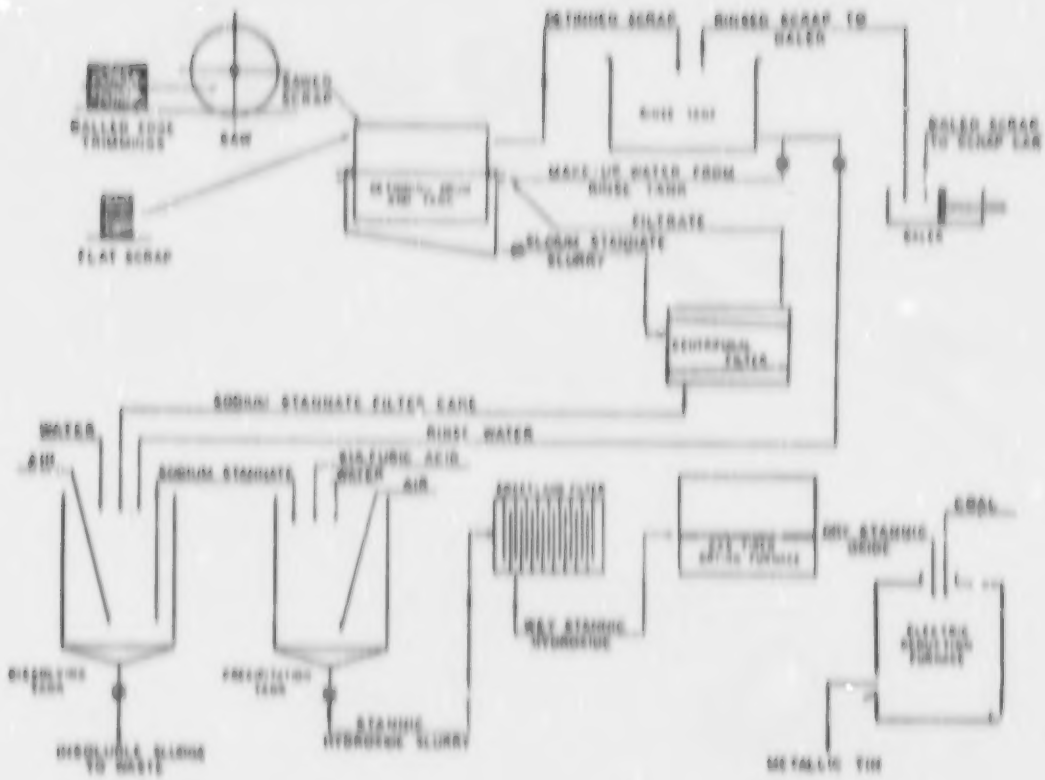

Figure 12A. Flow chart of the detinning and tin recovery process. (© Iron and Steel Engineer Year Book, 1953, page 207; used with permission.)

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IX-ENVIRONMENTAL ASPECTS

A. GENERAL INFORMATION

The most important environmental aspect of countercurrent rinsing is that the effluent from the countercurrent rinse tank has been concentrated to such an extent that direct or indirect recovery methods can be employed. Recovery of the effluent from the countercurrent rinse tanks can permit significant reductions in the loadings of the key pollutants such as tin, chlorides, fluorides, cyanides, suspended solids, etc., to the receiving stream. Since the most convenient method of recovery was the reuse of this effluent in the electrolytic process, this environmental study was accomplished with the effluent from the countercurrent rinse tanks recycled to the electrolytic plating solution tank.

It was the expressed intent of Weirton Steel Division to gather sufficient environmental background data on the original rinse system (see Figure 13) as to have a solid basis for comparison with the countercurrent rinse system (see Figure 14). In this regard, the environmental survey was designed to establish the existing average background loadings of key pollutants prior to the installation of the countercurrent rinsing system. These averages were then compared with the average loadings of the same parameters which were being diverted to the sewer after the installation of the countercurrent rinsing system. The background survey and the subsequent installation of the system shall be referred to as Phase I of the project in this report. The start-up and optimization of the system shall be referred to as Phase II and the evaluation of the system shall be referred to as Phase III of this project. During Phase I of the project, effluents from the recovery tank (Figures 13 and 15), spray wash (Figures 13 and 16), and hot rinse (Figures 13 and 17), were diverted to the sewer. During Phase III of the project only the effluents from the spray wash and the hot rinse were diverted to the sewer since the effluent from the countercurrent rinse tank was recycled to the plating solution tank.

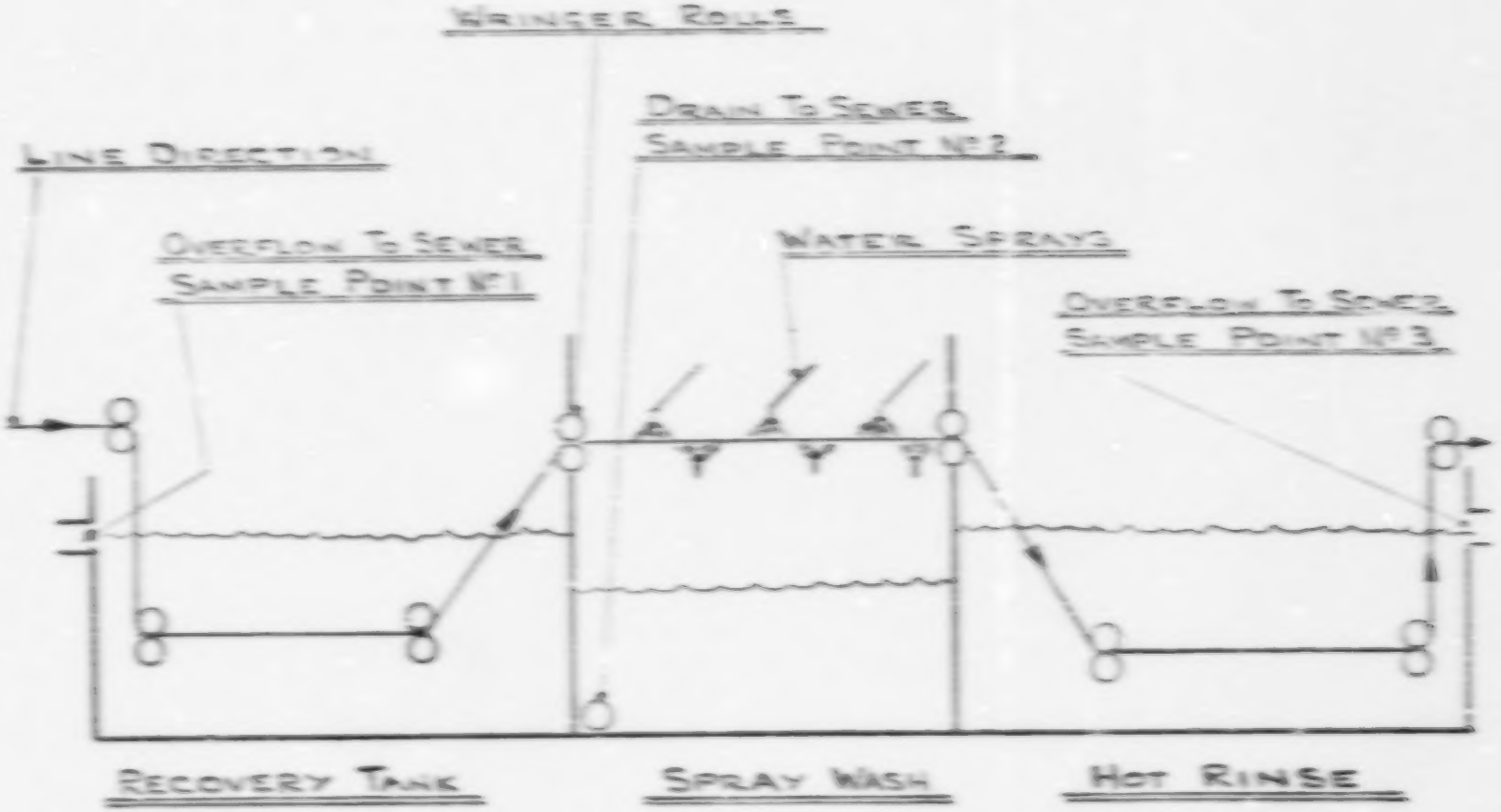

Figure 13. Background rinsing system, phase I sample points.

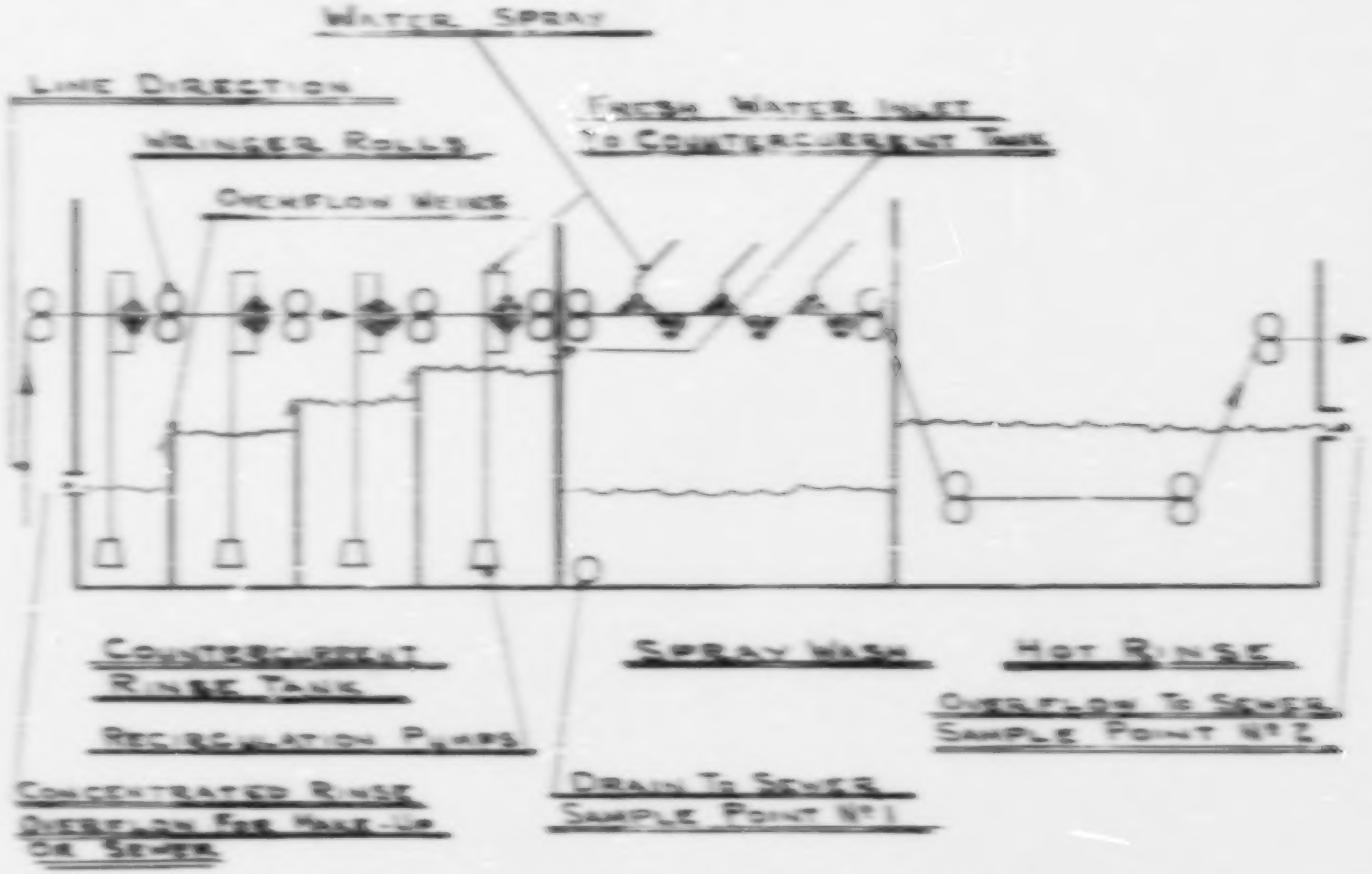

Figure 14. Countercurrent rinsing system, phase III sample points.

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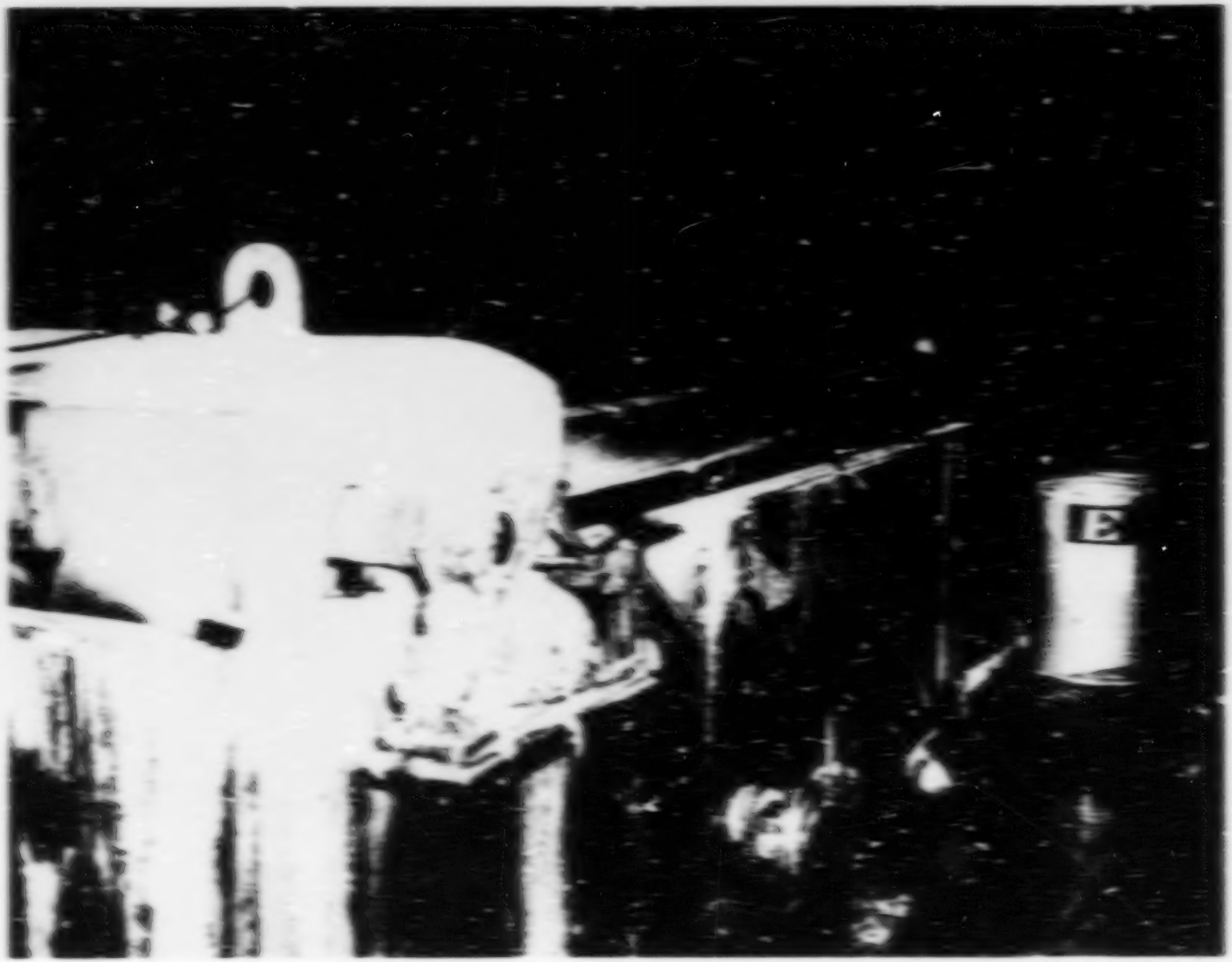

Figure 15. Recovery tank.

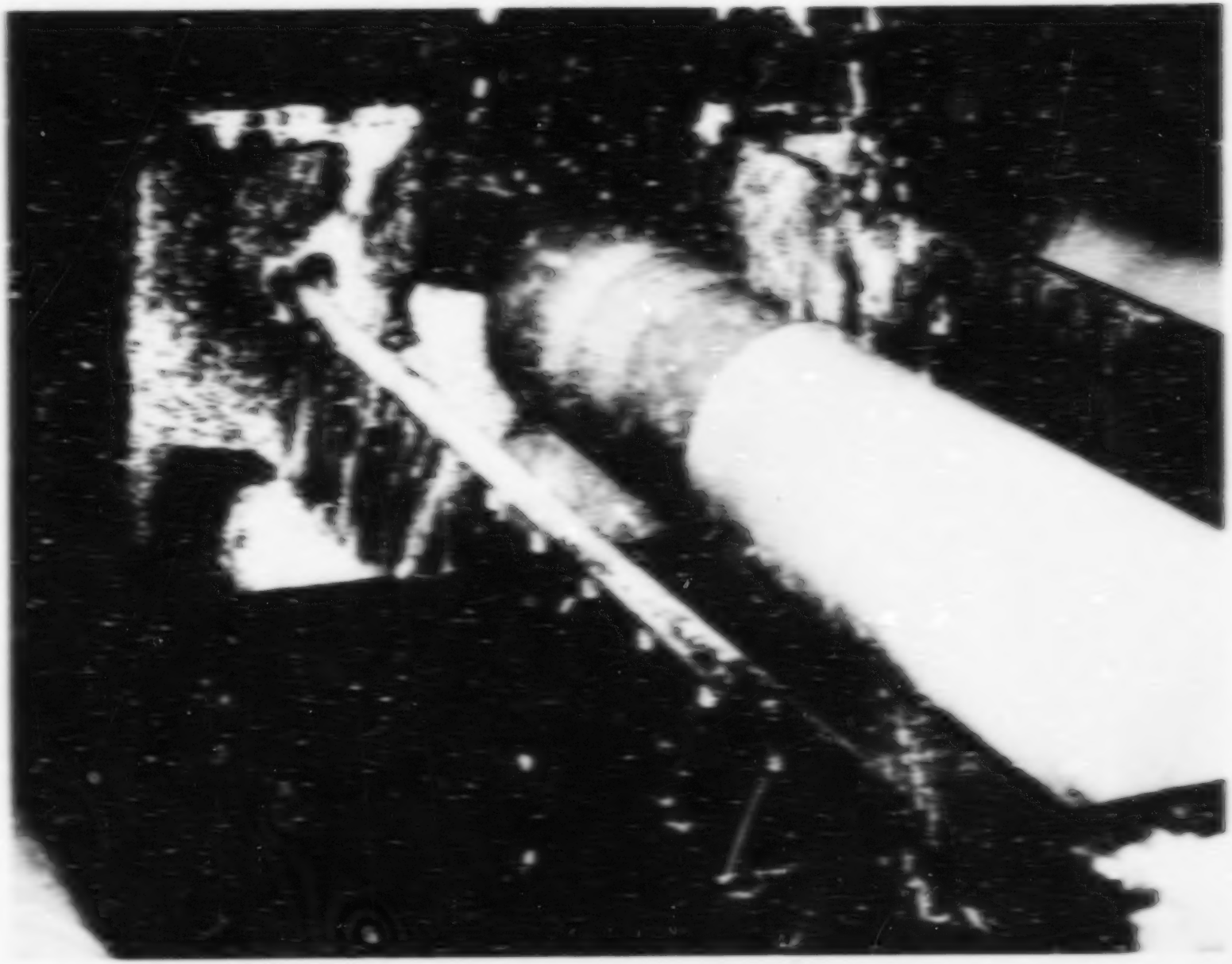

Figure 16. Spray wash.

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Figure 17. Hot rinse.

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B. BACKGROUND SURVEY (PHASE I) General Description

A total of seventeen samples was taken during the period of approximately one month (August 5 to September 6, 1974) to provide base line data with which to compare data obtained from the ensuing countercurrent rinse system. Figure 13 shows the basic rinse system and identifies the three sample locations. The recovery tank (Sample Point No. 1) was merely a dunk tank with an average incoming fresh water supply of approximately five gallons per minute. The spray wash (Sample Point No. 2) rinse section was composed of a series of sprays above and below the strip with an average flow of about fifty gallons per minute. The hot rinse (Sample Point No. 3) section was composed of a dunk tank which was occupied with hot water (180°F.) with a make-up flow of forty gallons per minute. The overflow or drains of each of the three rinse sections were channeled to the sewer during the base line period.

Figure 18 is a schematic of the basic sampling facilities. A flow meter was installed at each sampling location to measure the amount of fresh water coming into each rinse section. The summation of these flows was representative of the total effluent from the plating process, including dragout and evaporation. The effluent from each rinse was channeled into a polypropylene sampling box. Two representative samples of the effluent were collected from each polypropylene box utilising the refrigerated pressure operating sampler described in Figure 19. One sample was treated with sodium hydroxide to preserve cyanides and the other sample was left untreated for the remainder of the analyses. The background samples were then analysed for the following parameters:

Temperature	Suspended Solids
pH	Cadmium
Acidity or Alkalinity	Chromium
Chlorides	Iron
Chemical Oxygen Demand	Nickel
Free Cyanides	Phosphates
Total Cyanides	Potassium
Fluorides	Sodium
Sulfates	Tin

All samples analysed were collected as 24-hour composites.

Flow Rates

During the background survey all flows were obtained by setting the make-up water to the desired level using calibrated flow meters. These flow meters provided an instantaneous read-out and also provided an electronic signal to the integrators which totalised the flows. Due to problems encountered with the integrators, the instantaneous read-out was checked periodically and the daily flow calculated from the direct read-out. The flow meters were calibrated periodically using the stopwatch and bucket technique.

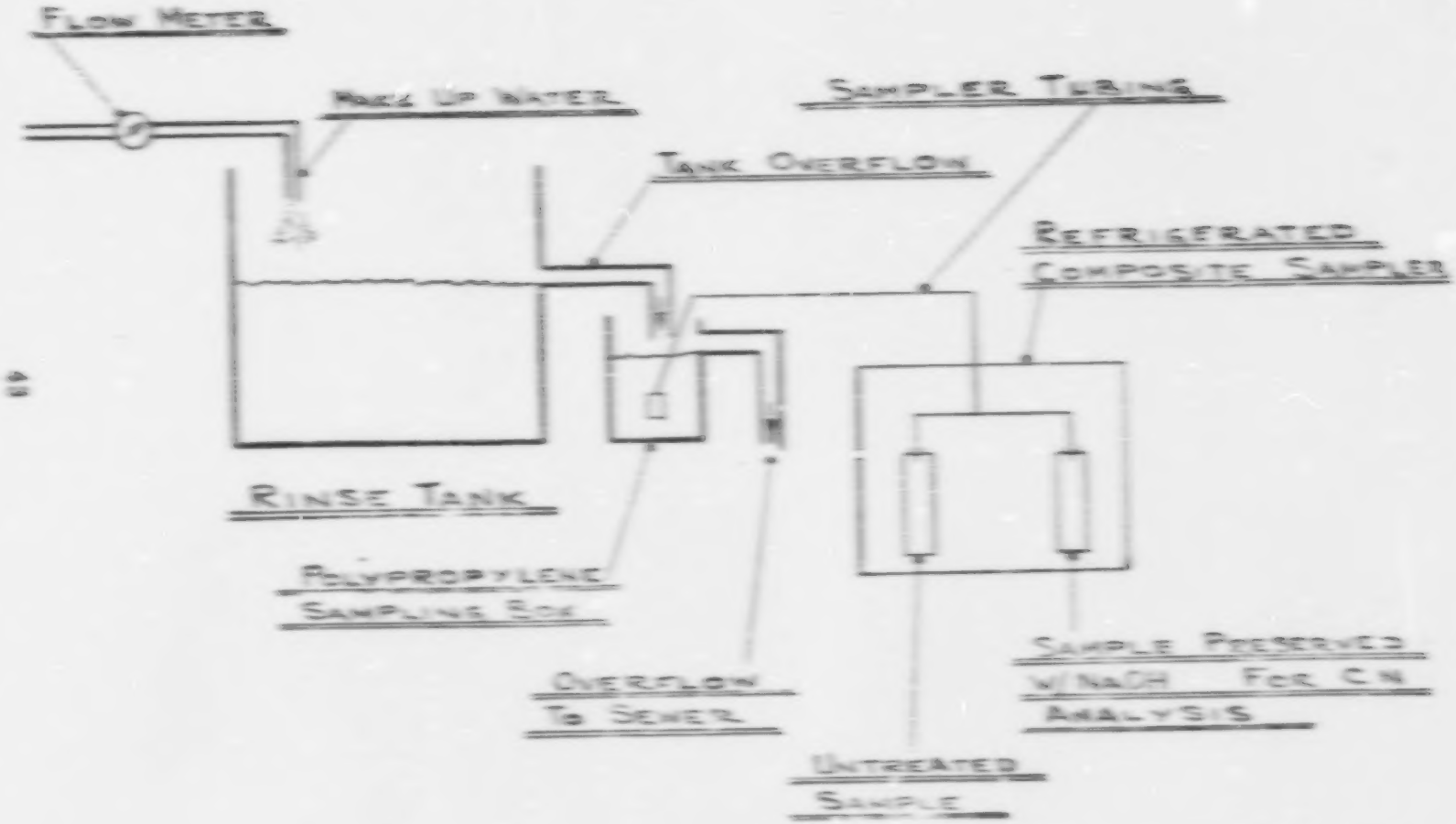
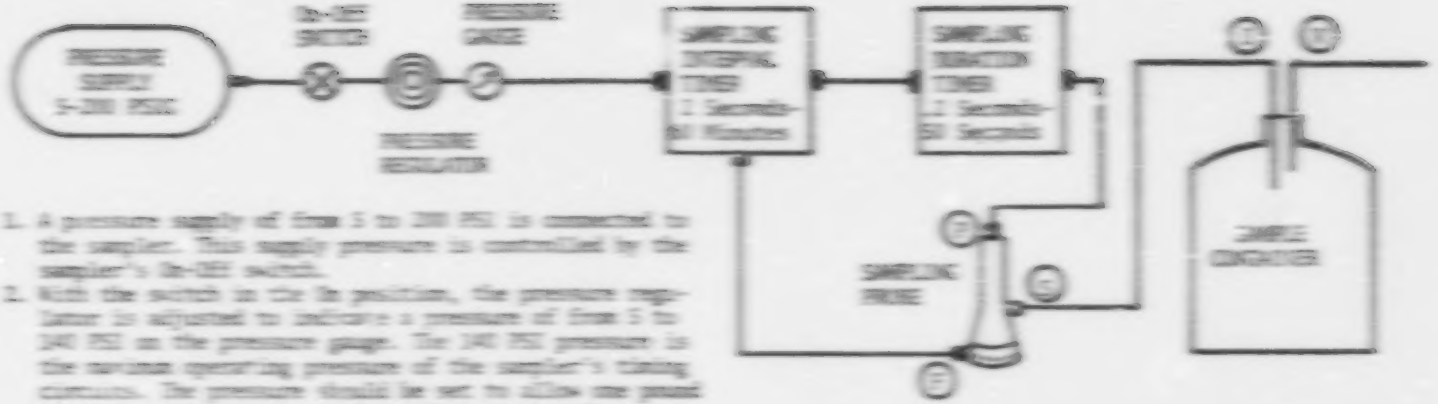


Figure 16. Sampling diagram essentially the same for: recovery tank, spray wash, hot rinse1.



1. A pressure supply of from 1 to 200 PSI is connected to the sampler. This supply pressure is controlled by the sampler's On-Off switch.
2. With the switch in the On position, the pressure regulator is adjusted to indicate a pressure of from 1 to 140 PSI on the pressure gauge. The 140 PSI pressure is the maximum operating pressure of the sampler's timing circuits. The pressure should be set to allow one pound of pressure for every two feet of vertical lift.
3. Pressure is now applied to the sampling interval timer. This timer controls the length of time between samples. The interval timer is continually adjustable from 1 seconds to 61 minutes between samples.
4. When the interval timer completes its time cycle it applies pressure to the sampling duration timer. This timer controls the length of time that the sampler releases pressure to the sampling probe. The greater the vertical lift or the longer the horizontal run, the longer the period of time that is required to purge the sample from the sample tubing.
5. Upon completion of the duration timer's time cycle, the applied pressure is released and enters connection "P" on the sampling probe. The sampling probe is a gravity-fill unit and holds a sample of 10 ml. Pressure is exerted upon the liquid contained in the probe and forces it to exit through the fitting "S" located on the side of the probe. The liquid travels through the connecting tubing and into the sample container. The sample container may be located at any point from several feet to over 200 feet from the sampling probe. Usually the container is located in the sampler's case or refrigerator.
NOTE: Only the sampling probe is submerged in the liquid to be sampled. The liquid never enters the sampler's control circuits.
6. When the sample is deposited in the sample container, the pressure in the lines is vented through the "P" connection on the sample container. In the event that the total composite sample taken over a period of time should exceed the container's capacity, it will be discharged through the "P" connection on the container.
7. After the sampling lines are purged of all vestige, the timing circuits next reset themselves automatically. During this reset period which occurs immediately after the sample is taken, the timers next reset their timing valves. The vent pressure from the timing valve on the interval timer is connected to the sampling probe through connection "P". This vent pressure is used to purge the probe's inlet screen of any foreign matter.
8. The sampler requires a pressure supply of from 1 to 200 PSI. N-12, nitrogen or compressed air can be used to power the sampler. Since the sampler is operated on pressure only, it is explosion-proof.

Figure 18. Schematic, pressure operated sampler.

Chemical Analysis

The analyses were performed using procedures found in one of these three sources:

1. Standard Methods for the Examination of Water and Wastewater, 15th Edition;
2. ASTM Standards, Part 23, Water; Atmospheric Analysis;
3. E.P.A. Manual of Methods for Chemical Analysis of Water and Wastes.

The only exception of the above statement pertains to the analysis of free cyanides. Since an approved method has not been published, an American Iron and Steel Institute's method was used. A summary of each procedure used for each parameter is listed in Appendix I (4, 5, 6). All solids were analyzed on a Perkin-Elmer Model 306 Atomic Absorption Spectrophotometer.

Results

Table 2 indicates the average daily loading in pounds for each final rank over the seventeen-day sampling duration. A tally tabulation of the results can be found in Appendix II.

TABLE II

CHART I - BACKGROUND EFFLUENT LOADINGS

	RECOVERY TANK Average Effluent Loading		SPRAY WASH Average Effluent Loading		HOT RINSE Average Effluent Loading	
Flow	5 gpm = 7200 gpd		53.53 gpm = 77,082 gpd		40 gpm = 57,600 gpd	
Temperature	112°F.		93°F.		102°F.	
pH	4.45		4.14		5.31	
Acidity/ Alkalinity	Acidity/7.99	lbs/Day	Acidity/38.1	lbs/Day	Alkalinity/1.93	lbs/Day
Chlorides	153	lbs/Day	82.9	lbs/Day	35.7	lbs/Day
C.O.D.	121	lbs/Day	27.2	lbs/Day	17.7	lbs/Day
Free Cyanides	.032	lbs/Day	0.15	lbs/Day	.045	lbs/Day
Total Cyanides	2.35	lbs/Day	0.66	lbs/Day	.14	lbs/Day
Fluorides	281	lbs/Day	59.6	lbs/Day	13.0	lbs/Day
Sulfates	17.5	lbs/Day	113	lbs/Day	75.3	lbs/Day
Suspended Solids	21.6	lbs/Day	56.9	lbs/Day	15.0	lbs/Day
Chromium	0.051	lbs/Day	0.015	lbs/Day	Detectable Limit	
Iron	3.86	lbs/Day	1.40	lbs/Day	0.27	lbs/Day
Nickel	0.044	lbs/Day	≤Detectable Limit		≤Detectable Limit	
Potassium	0.47	lbs/Day	3.09	lbs/Day	1.97	lbs/Day
Sodium	260	lbs/Day	94.4	lbs/Day	16.9	lbs/Day
Tin	203	lbs/Day	58.6	lbs/Day	3.25	lbs/Day

6. EVALUATION OF COUNTERCURRENT RINSING (PHASE III)

General Description

After the start-up and optimisation of the system (Phase II), a series of eleven samples were taken over a period of six weeks August 26 to October 3, 1973) to evaluate the improvement in pollutant loadings coming from the new rinse system featuring a countercurrent rinse tank as the primary rinse. (See Figure 14 for a schematic of the countercurrent rinsing system.) As can be seen from Figure 14, the spray wash and the hot rinse were being used as make-up for the plating solution. The identical sample points (Figure 14) and sampling facilities (Figure 18) were used for both Phase I (Background) and Phase III (Countercurrent Evaluation) sampling programs. Sampling of the countercurrent system was delayed several times due to equilibrium problems in the plating process.

Flow Rates

Due to problems which developed with the flow meters, the flows which were obtained during the countercurrent rinsing system evaluation were arrived at by the stopwatch and bucket technique. The flows were set before each test interval and re-checked at the end of the 24-hour testing period to insure their reliability.

Chemical Analysis

The chemical analyses of the effluents from the spray wash and the hot rinse were analysed by the same procedures as previously outlined. Summaries of the analytical methods may be found in Appendix I.

Results

Table III indicates the average daily loading, in pounds, of the eleven samples taken for the countercurrent rinse system evaluation. A complete day-by-day tabulation of the analysis may be found in Appendix III.

TABLE III

STAGE III - COUNTERCURRENT RINSING EFFLUENT LOADING

	SPRAY WASH Average Effluent Loadings	HOT RINSE Average Effluent Loadings
Flow	27,027 g.p.d.	46,172 g.p.d.
Temperature	88.3 Deg. F.	172.2 Deg. F.
pH	5.3	7.5
Acidity/Alkalinity	1.28 lbs. Alkalinity	7.41 lbs. Alkalinity
Chlorides	10.7 lbs	92.3 lbs.
C.O.D.	1.91 lbs.	2.38 lbs.
Free Cyanides	0.014 lbs.	0.035 lbs.
Total Cyanides	0.040 lbs.	0.065 lbs.
Fluorides	7.45 lbs.	0.99 lbs.
Sulfates	27.0 lbs.	42.9 lbs.
Suspended Solids	10.1 lbs.	7.80 lbs.
Chromium	≤ Detectable Limit	≤ Detectable Limit
Iron	0.16 lbs.	0.33 lbs.
Nickel	≤ Detectable Limit	≤ Detectable Limit
Potassium	1.05 lbs.	1.64 lbs.
Sodium	11.6 lbs.	5.81 lbs.
Tin	11.0 lbs.	2.52 lbs.

V. DISCUSSION OF RESULTS

Table IV illustrates the magnitude of reduction of the major pollutants that can be achieved by one hundred percent recycle of the effluent from a countercurrent rinsing system on a high speed electrolytic tin plating operation. Total recovery of the countercurrent rinsing effluent occurred during the two months preceeding the evaluation until approximately six weeks after the termination of the evaluation.

An analysis of the analytical results from this survey indicates the following reduction in daily loadings of key pollutant from an electrolytic tin plating operation when a countercurrent rinsing recovery system is employed:

Tin	95% Reduction
Total Cyanides	97% Reduction
Fluorides	98% Reduction
Chlorides	88% Reduction
Sodium	95% Reduction

During this period in which the effluent from the countercurrent rinse tank was completely re-used as make-up for the plating solution the possible environmental improvements were demonstrated. However, as indicated in Section VII of this report, product quality problems prevented the continued operation of the electrolytic plating line utilising this concept. In this regard, three alternatives have been proposed in Section VIII of this report, which could eliminate the product quality problems previously described. The environmental aspects of each alternative is discussed below:

1. Use of Hydrofluoric Acid - Section VIII-A describes the mechanism by which this alternative should eliminate product quality problems. The utilisation of hydrofluoric acid as a source for fluoride and to control pH should permit the continuous recycle of one hundred percent of the countercurrent rinsing effluent into the plating solution tank, thus achieving the reductions in pollutant discharges indicated above. However, a potential safety hazard exists in the handling of hydrofluoric acid, and the acceptance of this alternative is questionable even if the safety precautions described in Section VIII-A are adopted.

2. Use of an Alkalinity Removal Unit - A description of the operating principles of an alkalinity removal unit and its ability to eliminate the product quality problems associated with this project is documented in Section VIII-B. This unit would treat the countercurrent rinse tank effluent, utilizing electrodialysis principles, and it should permit the recycle of one hundred percent of the countercurrent rinsing effluent into the plating solution tank, thus achieving the pollutant reductions previously cited. A small quantity of sodium hydroxide would be produced as a by-product from the alkalinity removal treatment process. This material would be utilized in a wastewater neutralization treatment system serving the Tin Mill operations.

3. Indirect Recycle Via Detinning Plant - As stated in Section VIII-C, Wolfson Steel Division has a unique advantage in that the company operates a Detinning Plant which has the capability to remove the tin from the countercurrent tank effluent. By storing the effluent which cannot be directly re-used and transporting it to the Detinning Plant, a significant amount of the pollutants from the tin plating operations can be recovered and thus preventing their return to the environment. If zero discharge from the tin plating system cannot be achieved, then tests should be conducted to find out exactly how much blowdown must be removed from the system to prevent the accumulation of materials in untenable amounts. If, for example, it can be demonstrated that the system will work satisfactorily with a 70 percent recycle, the remaining 30 percent could be transported to the Detinning Plant for recovery. This alternative should also achieve the reduction in pollutant loadings previously documented.

This project has shown that significant environmental benefits can be derived by the direct or indirect recovery of the countercurrent rinsing effluent. The adoption of any one of the three proposed alternative solutions for the product quality problem should accomplish the desired environmental benefits. The selection of the alternative to be utilized therefore, requires one of economic, process, and safety considerations.

TABLE IV

COMPARISON OF BACKGROUND VS COUNTERCURRENT RINSING EFFLUENT LOADINGS

	Background Evaluation Total Average Effluent Loading	Countercurrent Rinsing Evaluation Total Average Effluent Loading	% Reduction
Flow	-	-	-
Temperature	-	-	-
pH	-	-	-
Acidity/Alkalinity	88.2 lbs. Acidity	8.69 lbs Alkalinity	-
Chlorides	271.6 lbs.	33.0 lbs.	87.8
C.O.D.	165.9 lbs.	6.19 lbs.	96.3
Free Cyanides	0.237 lbs.	.069 lbs.	70.9
Total Cyanides	3.15 lbs.	.105 lbs.	96.7
Fluorides	359.6 lbs.	8.64 lbs.	97.6
Sulfates	202.8 lbs.	69.8 lbs.	65.6
Suspended Solids	93.5 lbs.	17.9 lbs.	80.9
Chromium	0.066 lbs.	≤ Detectable Limit	-
Iron	5.53 lbs.	.49 lbs.	91.1
Nickel	0.066 lbs.	≤ Detectable Limit	-
Potassium	5.53 lbs.	2.69 lbs.	51.4
Sodium	371.3 lbs.	17.41 lbs.	95.3
Tin	270 lbs.	13.52 lbs.	95.0

A-BIBLIOGRAPHY

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3. Krombiels, A. J., "Recovery of Tin from Scrap Tinplate," Iron and Steel Engineer Yearbook, 1953, p. 206.

4. 1974 Manual and Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, p. 239.

5. Standard Methods for the Examination of Water and Wastewater, 13th Edition, p. 495, Method 220 (1971).

6. ASTM Standards, Part 28, Water; Atmospheric Analysis, p. 273, Method 512-67, Referee Method A (1973).

APPENDIX NO. 1

ANALYTICAL METHODS

pH - All pH measurements were made on a Beckman Zeromatic II using glass electrode with a saturated calomel electrode used as the reference potential as specified in the 1974 Manual of Methods for Chemical Analysis of Water and Wastes, U.S Environmental Protection Agency; Page 239. The instrument was calibrated daily with buffer solutions.

ACIDITY - All acidity analyses were performed as outlined by the procedure listed in Standard Methods for the Examination of Water and Wastewater, 13th Edition, Page 50, Method 10. (1971).

All acidity analyses were performed by titrating an unaltered sample to a pH of 4.5 using standardized sodium hydroxide as the titrant.

ALKALINITY - All alkalinity analyses were performed as outlined by procedure listed in Standard Methods for the Examination of Water and Wastewater, 13th Edition, Page 52, Method 108 (1971).

All alkalinity analyses were performed by titrating an unaltered sample to a determined end point of pH 4.5 using standardized sulfuric acid as a titrant.

CHEMICAL OXYGEN DEMAND (C.O.D.) - All C.O.D. analyses were performed as outlined by the procedure listed in Standard Methods for the Examination of Water and Wastewater, 13th Edition, Page 495, Method 220 (1971). A proper aliquot of sample is mixed with sulfuric acid, mercuric sulfate, silver sulfate, and standard potassium dichromate and the excess potassium dichromate in the cooled mixture is titrated with standard ferrous ammonium sulfate to the endpoint indicated by ferroin.

CHLORIDES 1 - For the Quray Wash and Hot Pipes Locations - all chloride analyses for the above locations were performed according to the procedure outlined in the ASTM Standards, Part 23, Water; Atmospheric Analysis, Page 271, Method D12-67, Reapproved Method A (1973). According to this method, an acidified sample is titrated with a dilute mercuric nitrate solution to a blue-violet endpoint shown by a mixed diphenylcarbazone-bromophenol blue indicator.

2 - For the Recovery Tank and the Countercurrent Tank Locations - because of interferences shown when using the above method, pre-treatment steps, as outlined in Standard Methods for the Examination of Water and Wastewater,

CN- cont'd (cont'd) - 13th edition, Page 379, Method 303C, 48 (1971) were followed. This pre-treatment involves boiling the acidified sample to remove volatile compounds and then oxidizing the sample with hydrogen peroxide. After the pre-treatment, the standard mercuric nitrate procedure (as outlined in the preceding paragraph) is followed.

CYANIDES

1 - Total - all total cyanide analyses were performed as outlined in Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, Page 40 (1974). All samples were collected by a refrigerated sampler and split into a treated and untreated plastic bottles. The treated bottles contained sodium hydroxide to prevent significant losses in cyanide.

The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux system of mineral acid in the presence of mercuric chloride and magnesium chloride and a distillation operation where the HCN is then collected in a scrubber containing sodium hydroxide solution. The pH of the scrubber solution is adjusted to a pH of 7.0 and reacted with chloramine - T. Proper sample aliquots were used in order that all samples could be determined colorimetrically at a wave length of 620 nm.

2 - Free - all free cyanide analyses were performed by the same procedure as that listed for total cyanides except that the catalysts (mercuric and magnesium chlorides) were not added, thus preventing the breakdown of the iron-cyanide complexes. The samples were also preserved by both refrigeration and by the addition of sodium hydroxide.

FLUORIDES - all the fluoride analyses were determined potentiometrically using a selective ion fluoride electrode in conjunction with a standard single junction sleeve-type reference electrode and a pH meter having an expanded millivolt scale. The procedure used is contained in manual of Methods for Chemical Analysis of Water and Wastes, Page 83 (1974), published by the Environmental Protection Agency. The method involves adjusting the pH of the sample by means of a buffer solution and chelating the polyvalent cations which could interfere.

PHOSPHORUS - All phosphorus analyses were performed according to the method outlined in a manual of Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, Page 349. Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

SULFATES - All sulfate analyses were performed as outlined in Standard Methods for the Examination of Water and Wastewater, 13th Edition, Page 334, Method 156C (1971). Sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a spectrophotometer and compared to a curve prepared from standard sulfate solutions.

METALS - (Cadmium, chromium, iron, nickel, potassium, sodium and tin) All the metals were treated and analyzed as outlined in the manual of Methods for Chemical Analysis of Water and Wastes, Environmental Protection Agency Metals (Atomic Absorption), Page 78. All metal samples were preserved with HNO_3. All analyses were performed on a Perkin-Elmer Model 306 Atomic Absorption Spectrophotometer. The samples were prepared by taking an appropriate aliquot to which HNO_3 had been added and evaporating carefully to dryness. Repeated HNO_3 additions were made until the removal of organic material was completed. The samples were then redissolved and analyzed using the atomic absorption spectrophotometer equipped with hollow cathode lamps.

APPENDIX II

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES

DATE 8/5 to 8/6/74

	RECOVERY TANK	DIPRAY WASH	HOT RINSE
FLOW TO SEWER	5gpm = 7,200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	110° F	93°F	181°F
pH	4.5	4.0	3.8
ACIDITY/ALKALINITY	0ppm = 0 lbs	Acidity 40 ppm = 17.7 lbs	Acidity 8.0 ppm = 3.84 lbs
CHLORIDES	1800 ppm = 108. lbs/Day	140 ppm = 58.4 lbs/Day	114 ppm = 54.8 lbs/Day
COD	892 ppm = 53.6 lbs/Day	33 ppm = 139. lbs/Day	41 ppm = 19.7 lbs/Day
FREE CYANIDES	1.44 ppm = .09 lbs/Day	------	------
TOTAL CYANIDES	54 ppm = 3.24 lbs/Day	.54 ppm = .23 lbs/Day	.038 ppm = .018 lbs/Day
FLUORIDES	4,200 ppm = 252 lbs/Day	86 ppm = 36.1 lbs/Day	46 ppm = 22. lbs/Day
SULFATES	250 ppm = 15.0 lbs/Day	166 ppm = 69.8 lbs/Day	166 ppm = 79.7 lbs/Day
SUSPENDED SOLIDS	694 ppm = 41.7 lbs/Day	138 ppm = 580.0 lbs/Day	18 ppm = 8.65 lbs/Day
CHROMIUM	0.86 ppm = .048 lbs/Day	0.03 ppm = .013 lbs/Day	*
IRON	144.5 ppm = 8.68 lbs/Day	2.4 ppm = 1.01 lbs/Day	.40 ppm = .19 lbs/Day
NICKEL	1.25 ppm = .073 lbs/Day	*	*
POTASSIUM	7.0 ppm = 0.42 lbs/Day	4.78 ppm = 2.01 lbs/Day	4.0 ppm = 1.92 lbs/Day
SODIUM	3517 ppm = 211 lbs/Day	132 ppm = 55.5 lbs/Day	23.3 ppm = 11.2 lbs/Day
ZINC	3180 ppm = 191 lbs/Day	94 ppm = 39.5 lbs/Day	18.0 ppm = 8.6 lbs/Day

* < Detectable Limit

------ = Analysis Not Performed

COUNTERJURRENT RINSING
PHASE I - CHEMICAL ANALYSES DATE 8/6 to 8/7/81

	RECOVERY TANK	STRAX WASH	HOT RINSE
FLOW TO DEVCO	5 gpm = 7200 gpd	35gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	120° F	92°F	185°F
pH	4.4	4.2	4.6
ACIDITY/ALKALINITY	Acidity 20 ppm = 1.20 lbs/Day	Acidity 18 ppm = 5.04 lbs/Day	Alkalinity 6.0 ppm = 2.76 lbs/Day
CHLORIDES	2290 ppm = 135 lbs/Day	85 ppm = 35.7 lbs/Day	72 ppm = 34.6 lbs/Day
COD	1,35 ppm = 76.3 lbs/Day	27 ppm = 11.3 lbs/Day	46 ppm = 22.1 lbs/Day
FREE CYANIDES	0.40 ppm = .024 lbs/Day	0.84 ppm = .35 lbs/Day	======
TOTAL CYANIDES	137 ppm = 8.23 lbs/Day	3.52 ppm = 1.48 lbs/Day	.35 ppm = .077 lbs/Day
FLUORIDES	5500 ppm = 330 lbs/Day	96.0 ppm = 53.5 lbs/Day	56 ppm = 26.9 lbs/Day
SULFATES	312 ppm = 18.7 lbs/Day	166 ppm = 69.8 lbs/Day	158 ppm = 75.9 lbs/Day
SUSPENDED SOLIDS	625 ppm = 37.5 lbs/Day	98 ppm = 41.2 lbs/Day	11 ppm = 5.28 lbs/Day
CHROMIUM	0.95 ppm = .057 lbs/Day	*	*
IRON	89.5 ppm = 5.37 lbs/Day	1.6 ppm = 0.67 lbs/Day	0.40 ppm = .19 lbs/Day
NICKEL	0.80 ppm = .048 lbs /Day	*	*
POTASSIUM	7.25 ppm = .44 lbs/Day	4.16 ppm = 1.79 lbs/Day	3.71 ppm = 1.78 lbs/Day
SODIUM	4390 ppm = 264 lbs/Day	86 ppm = 36.1 lbs/Day	84.4 ppm = 41.7 lbs/Day
TIN	3775 ppm = 227 lbs/Day	52 ppm = 21.9 lbs/Day	9 ppm = 4.32 lbs./Day

* = <Detectable Limit

====== = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES

DATE 8/7 to 8/8/74

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO DEMIN	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	111°F	93° F	102° F
pH	4.65	4.3	4.15
ACIDITY/ALKALINITY	Alkalinity 506 ppm = 17.8 lbs/Day	Acidity 12.0 ppm = 5.04 lbs/Day	Acidity 4.0 ppm = 1.92 lbs/Day
CHLORIDES	4400 ppm = 132 lbs/Day	185 ppm = 77.8 lbs/Day	156 ppm = 74.9 lbs/Day
COD	1370 ppm = 83.5 lbs/Day	46.5 ppm = 17.9 lbs/Day	39 ppm = 18.7 lbs
FREE CYANIDES	0.41 ppm = .025 lbs/Day	0.94 ppm = .40 lbs/Day	*****
TOTAL CYANIDES	120 ppm = 7.31 lbs/Day	2.4 ppm = 1.01 lbs/Day	0.21 ppm = 0.10 lbs/Day
FLUORIDES	8000 ppm = 305 lbs/Day	115 ppm = 48.3 lbs/Day	80 ppm = 38.0 lbs/Day
SULFATES	587 ppm = 17.2 lbs/Day	172 ppm = 72.3 lbs/Day	146 ppm = 70.14 lbs/Day
SUSPENDED SOLIDS	649 ppm = 39.0 lbs/Day	181 ppm = 76.1 lbs/Day	14 ppm = 6.73 lbs/Day
CHROMIUM	0.85 ppm = 0.05 lbs/Day	.02 ppm = .01 lbs/Day	*
IRON	74.5 ppm = 4.47 lbs/Day	2.5 ppm = 1.05 lbs/Day	0.39 ppm = .19 lbs/Day
NICKEL	0.65 ppm = .039 lbs/Day	*	*
POTASSIUM	7.7 ppm = 0.46 lbs/Day	4.5 ppm = 1.89 lbs/Day	3.78 ppm = 1.75 lbs/Day
SODIUM	4190 ppm = 249 lbs/Day	161 ppm = 67.7 lbs/Day	86.4 ppm = 41.7 lbs/Day
TIN	3785 ppm = 227 lbs/Day	83 ppm = 34.9 lbs/Day	11 ppm = 5.28 lbs/Day

* <Detectable Limit
***** Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSIS DATE 8/11 to 8/12/

	RECOVERY TANK	DIRAY WASH	HOT RINSE
FLOW TO PUMP	5 gpm = 3200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	107°F	95°F	176°F
pH	4.5	5.0	5.1
ACIDITY/ALKALINITY	0 ppm = 0 lbs/Day	ACIDITY 18 ppm = 7.57 lbs/Day	ACIDITY 4.0 ppm = 1.92 lbs/Day
CHLORIDES	1,511 ppm = 92.5 lbs/Day	165 ppm = 69.4 lbs/Day	96 ppm = 46.0 lbs/Day
COD	12,108 ppm = 607 lbs/Day	69 ppm = 29.0 lbs/Day	64 ppm = 30.7 lbs/Day
TOTAL CYANIDES	.85 ppm = .05 lbs/Day	======	.103 ppm = .05 lbs/Day
TOTAL CYANIDES	5.32 ppm = .32 lbs/Day	.564 ppm = .24 lbs/Day	.677 ppm = .33 lbs/Day
FLUORIDES	3581 ppm = 210 lbs/Day	88 ppm = 37.0 lbs/Day	14 ppm = 6.73 lbs/Day
SULFATES	233 ppm = 14.0 lbs/Day	180 ppm = 75.7 lbs/Day	154 ppm = 74.0 lbs/Day
SUSPENDED SOLIDS	1738 ppm = 74.3 lbs/Day	178 ppm = 52.1 lbs/Day	31 ppm = 14.9 lbs/Day
CHROMIUM	0.90 ppm = .056 lbs/Day	.09 ppm = .013 lbs/Day	<
IRON	101 ppm = 6.08 lbs/Day	3.7 ppm = 1.35 lbs/Day	0.33 ppm = .16 lbs/Day
NICKEL	0.55 ppm = .057 lbs/Day	<	<
POTASSIUM	6.5 ppm = 0.39 lbs/Day	4.84 ppm = 1.98 lbs/Day	4.09 ppm = 1.96 lbs/Day
BORIUM	3150 ppm = 189 lbs/Day	187 ppm = 87.9 lbs/Day	32.2 lbs = 15.5 lbs/Day
TIN	2531 ppm = 150 lbs/Day	87 ppm = 39.1 lbs/Day	15 lbs = 7.21 lbs/Day

< = Detectable Limit
====== = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES

DATE 8/13 to 8/16/76

	RECOVERY TANK	DIPAY WASH	HOT RINSE
FLOW TO SEWER	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	45 gpm = 97,660 gpd
TEMPERATURE	113°F	91°F	180°F
pH	4.5	4.8	5.4
ACIDITY/ALKALINITY	0.0 ppm = 0 lbs/Day	Acidity 96 ppm = 40.4 lbs/Day	Alkalinity 4.0 ppm = 1.92 lbs/Day
CHLORIDES	9200 ppm = 174 lbs/Day	860 ppm = 103 lbs/Day	72 ppm = 34.6 lbs/Day
COD	1588 ppm = 95.4 lbs/Day	93 ppm = 32.1 lbs/Day	32 ppm = 15.4 lbs/Day
FREE CYANIDES	.54 ppm = .032 lbs/Day	-------	.06 ppm = .029 lbs/Day
TOTAL CYANIDES	3.48 ppm = .15 lbs/Day	.62 ppm = .18 lbs/Day	.022 ppm = .068 lbs/Day
FLUORIDES	7015 ppm = 420 lbs/Day	151 ppm = 52.2 lbs/Day	15 ppm = 7.03 lbs/Day
SULFATES	-------	140 ppm = 75.7 lbs/Day	150 ppm = 72.1 lbs/Day
SUSPENDED SOLIDS	238 ppm = 14.29 lbs/Day	154 ppm = 53.1 lbs/Day	23 ppm = 10.3 lbs/Day
CHROMIUM	1.0 ppm = .06 lbs/Day	.03 ppm = .013 lbs/Day	*
IRON	72 ppm = 4.32 lbs/Day	3.2 ppm = 1.35 lbs/Day	.37 ppm = 0.18 lbs/Day
NICKEL	.93 ppm = .057 lbs/Day	*	*
POTASSIUM	8.65 ppm = .52 lbs/Day	4.71ppm= 1.98 lbs/Day	3.93 ppm = 1.87 lbs/Day
SODIUM	5900 ppm = 354 lbs/Day	209 ppm = 87.9 lbs/Day	35.7 ppm = 17.6 lbs/Day
TIN	4365 ppm = 261 lbs/Day	93 ppm = 39.1 lbs/Day	13 ppm = 6.24 lbs/Day

* = Detectable Limit

------- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES DATE 8/14 to 8/17/73

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO DEMER	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	111°F	90°F	180°F
pH	4.6	3.7	4.2
ACIDITY/ALKALINITY	Acidity 448 ppm = 8.53 lbs /Day	Acidity 84 ppm = 25.2 lbs	Acidity 4.0 ppm = 1.76 lbs /Day
CHLORIDES	1180 ppm = 191 lbs/Day	20 ppm = 35.3 lbs/Day	79 ppm = 38.0 lbs/Day
COD	1665 ppm = 86.8 lbs/Day	80 ppm = 33.6 lbs/Day	19 ppm = 9.13 lbs/Day
FREE CYANIDES	1.34 ppm = .08 lbs/Day	-------	.113 ppm = .05 lbs/Day
TOTAL CYANIDES	7.0 ppm = .42 lbs/Day	-------	.613 ppm = .27 lbs/Day
FLUORIDES	9400 ppm = 564 lbs/Day	155 ppm =65.2 lbs/Day	28 ppm = 13.5 lbs/Day
SULFATES	184 ppm = 11.0 lbs/Day	166 ppm =69.8 lbs/Day	150 ppm = 72.1 lbs/Day
SUSPENDED SOLIDS	193 ppm = 11.6 lbs/Day	175 ppm =73.6 lbs/Day	34 ppm = 16.3 lbs/Day
CHROMIUM	.75 ppm = .045 lbs/Day	.05 ppm =.02 lbs/Day	*
IRON	81.5 ppm = 4.89 lbs/Day	3.5 ppm =1.47 lbs/Day	0.5 ppm = .24 lbs/Day
NICKEL	1.00 ppm = .06 lbs/Day	*	*
POTASSIUM	10.45 ppm = .63 lbs/Day	4.89 ppm =2.06 lbs/Day	4.1 ppm = 1.98 lbs/Day
SODIUM	4500 ppm = 270 lbs/Day	205 ppm =86.2 lbs/Day	48.9 ppm = 23.5 lbs/Day
TIN	4795 ppm = 288 lbs/Day	150 ppm =63.1 lbs/Day	22 ppm = 10.6 lbs/Day

* = Detectable Limit

------- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES

DATE 8/19 to 8/20/74

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO TEMCO	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	105°F	91°F	183°F
pH	6.5	3.6	3.7
ACIDITY/ALKALINITY	Acidity 248 ppm = 14.9 lbs/Day	Acidity 190 ppm =63.1 lbs/Day	Acidity 16 ppm = 5.76 lbs/Day
CHLORIDES	3301 ppm = 198 lbs/Day	80 ppm = 33.6 lbs/Day	78 ppm = 37.5 lbs/Day
COD	1417 ppm = 85.1 lbs/Day	83 ppm = 34.9 lbs/Day	28 ppm =13.5 lbs/Day
FREE CYANIDES	.68 ppm = .041 lbs/Day	.31 ppm =.13 lbs/Day	.15 ppm = .072 lbs/Day
TOTAL CYANIDES	15 ppm = .90 lbs/Day	.51 ppm =.21 lbs/Day	.03 ppm = .14 lbs/Day
FLUORIDES	3800 ppm = 228 lbs/Day	240 ppm =101 lbs/Day	27 ppm =13.2 lbs/Day
SULFATES	357 ppm = 21.4 lbs/Day	170 ppm =72.2 lbs/Day	108 ppm = 52.1 lbs/Day
SUSPENDED SOLIDS	384 ppm = 23.1 lbs/Day	149 ppm =62.6 lbs/Day	47 ppm = 23.5 lbs/Day
CHROMIUM	.85 ppm = .051 lbs/Day	.03 ppm =.013 lbs/Day	*
IRON	78.5 ppm = 4.63 lbs/Day	5.3 ppm =2.23 lbs/Day	0.72 ppm = .35 lbs/Day
NICKEL	.85 ppm = .051 lbs/Day	*	*
POTASSIUM	8.55 ppm = .51 lbs/Day	5.36 ppm =2.25 lbs/Day	6.13 ppm = 2.98 lbs/Day
SODIUM	5400 ppm = 324. lbs/Day	372 ppm = 156 lbs/Day	46.7 ppm = 22.5 lbs/Day
Zinc	1820 ppm = 93.1 lbs/Day	289 ppm = 121 lbs/Day	31 ppm = 15.9 lbs/Day

* = <Detectable Limit

---- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSIS

DATE 8/[illegible] 8/[illegible]/[illegible]

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO SEWER	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	45 gpm = 57,600 gpd
TEMPERATURE	113°F	93°F	104°F
pH	4.5	3.9	6.5
ACIDITY/ALKALINITY	0 ppm = 0 lbs/Day	Acidity 138 ppm = 98.0 lbs/Day	0 ppm = 0 lbs/Day
CHLORIDES	1,020 ppm = 168 lbs/Day	384 ppm = 161 lbs/Day	92 ppm = 28.3 lbs/Day
COD	1533 ppm = 78.1 lbs/Day	100 ppm = 42.0 lbs/Day	16 ppm = 5.76 lbs/Day
FREE CYANIDES	******	******	0.0185
TOTAL CYANIDES	34 ppm = 1.72 lbs/Day	8.0 ppm = 3.36 lbs/Day	3.6 ppm = 1.15 lbs/Day
FLUORIDES	5800 ppm = 348 lbs/Day	245 ppm = 103 lbs/Day	37 ppm = 13.2 lbs/Day
SULFATES	330 ppm = 19.8 lbs/Day	176 ppm = 73.1 lbs/Day	135 ppm = 76.0 lbs/Day
SUSPENDED SOLIDS	267 ppm = 13.6 lbs/Day	112 ppm = 50.0 lbs/Day	39 ppm = 18.7 lbs/Day
CHROMIUM	0.80 ppm = .048 lbs/Day	.02 ppm = .008 lbs/Day	*
IRON	53.5 ppm = 3.01 lbs/Day	3.5 ppm = 1.43 lbs/Day	1.68 ppm = .33 lbs/Day
NICKEL	0.85 ppm = 0.051 lbs/Day	*	*
POTASSIUM	9.7 ppm = 0.52 lbs/Day	5.38 ppm = 2.32 lbs/Day	1.66 ppm = 2.03 lbs/Day
SODIUM	1200 ppm = 312 lbs/Day	341 ppm = 135 lbs/Day	48.9 ppm = 23.5 lbs/Day
TIN	3675 ppm = 221 lbs/Day	208 ppm = 75.0 lbs/Day	25 ppm = 12.0 lbs/Day

* = Detectable Limit

****** = Analysis not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSIS

DATE 8/21 to 8/22/74

	RECOVERY TANK	AIRAY WASH	HOT RINSE
FLOW TO DEMIN	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	110°F	92°F	103°F
pH	5.5	2.9	4.2
ACIDITY/ALKALINITY	Acidity 110 ppm = 6.07 lbs/Day	Acidity 130 ppm = 55.5 lbs/Day	Acidity 18 ppm = 8.6 lbs/Day
CHLORIDES	4800 ppm = 288 lbs/Day	460 ppm = 185 lbs/Day	144ppm = 69.2 lbs/Day
COD	6238 ppm = 375 lbs/Day	106 ppm = 44.6 lbs/Day	46 ppm = 22.1 lbs/Day
FREE CYANIDES	.633 ppm = .038 lbs/Day	.273 ppm = .115 lbs/Day	.087 ppm = .042 lbs/Day
TOTAL CYANIDES	2.5 ppm = .15 lbs/Day	.62 ppm = .27 lbs/Day	.174 ppm = .083 lbs/Day
FLUORIDES	5000 ppm = 300 lbs/Day	223 ppm = 94.6 lbs/Day	33 ppm = 15.9 lbs/Day
SULFATES	287 ppm = 17.2 lbs/Day	162 ppm = 68.1 lbs/Day	150 ppm = 72.1 lbs/Day
SUSPENDED SOLIDS	271 ppm = 16.3 lbs/Day	203 ppm = 85.3 lbs/Day	81 ppm = 38.9 lbs/Day
CHROMIUM	.85 ppm = .051 lbs/Day	*	*
IRON	67 ppm = 4.02 lbs/Day	5.2 ppm = 2.19 lbs/Day	.74 ppm = .36 lbs/Day
NICKEL	.93 ppm = .056 lbs/Day	*	*
POTASSIUM	8.85 ppm = .53 lbs/Day	5.39 ppm = 2.27 lbs/Day	6.30 ppm = 3.07 lbs/Day
SODIUM	6350 ppm = 381 lbs/Day	331 ppm = 139 lbs/Day	85.5 ppm = 41.7 lbs/Day
TIN	4655 ppm = 268 lbs/Day	271 ppm = 114 lbs/Day	36 ppm = 17.3 lbs/Day

* = Detectable Limit

---- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES

DATE 8/[illegible] to 8/[illegible]

	RECOVERY TANK	RINSE WASH	HOT RINSE
FLOW TO (EMCI)	5 gpm = 7200 gpd	35 gpm = 50,400 gpd	40 gpm = 57,600 gpd
TEMPERATURE	115°F	95°F	170°F
pH	4.5	3.6	4.0
ACIDITY/ALKALINITY	0 ppm = 0 lbs/Day	Acidity 104 ppm = 60.1 lbs/Day	Acidity 10 ppm = 4.8 lbs/Day
CHLORIDES	470 ppm = 270 lbs/Day	566 ppm = 800 lbs/Day	38 ppm = 18.3 lbs/Day
COD	1303 ppm = 752.2 lbs/Day	84 ppm = 35.3 lbs/Day	54 ppm = 25.9 lbs/Day
FREE CYANIDES	-------	.017 ppm = .14 lbs/Day	-------
TOTAL CYANIDES	17.6 ppm = 1.01 lbs/Day	2.3 ppm = .97 lbs/Day	.117 ppm = .06 lbs/Day
FLUORIDES	6031 ppm = 348 lbs/Day	320 ppm = 135 lbs/Day	33 ppm = 15.7 lbs/Day
SULFATES	384 ppm = 23.1 lbs/Day	150 ppm = 63.0 lbs/Day	150 ppm = 71.1 lbs/Day
SUSPENDED SOLIDS	249 ppm = 15.0 lbs/Day	170 ppm = 71.5 lbs/Day	81 ppm = 38.9 lbs/Day
CHROMIUM	.90 ppm = .054 lbs/Day	.02 ppm = .008 lbs/Day	*
IRON	71 ppm = 4.26 lbs/Day	5.5 ppm = 2.31 lbs/Day	.72 ppm = .35 lbs/Day
NICKEL	.55 ppm = .033 lbs/Day	*	*
POTASSIUM	9.1 ppm = .55 lbs/Day	5.51 ppm = 2.32 lbs/Day	4.17 ppm = 2.00 lbs/Day
SODIUM	6503 ppm = 390 lbs/Day	418 ppm = 176 lbs/Day	62.2 ppm = 29.9 lbs/Day
TIN	7930 ppm = 476 lbs/Day	347 ppm = 146 lbs/Day	37 ppm = 17.8 lbs/Day

* = Detectable limit

------- = Analysis Not Performed

COUNTERCURRENT RINSING

PHASE I - CHEMICAL ANALYSES

DATE 8/26 to 8/27/7[illegible]

	RECOVERY TANK	RINSE WASH	HOT RINSE
FLOW TO TANK	5 gpm = 7200 gpd	80 gpm = 115,200 gpd	50 gpm = 72,000 gpd
TEMPERATURE	110°F	96°F	100°F
pH	1.1	4.2	6.5
ACIDITY/ALKALINITY	485 ppm = 29.1 lbs/Day	ACIDITY 4.40 ppm =3.70 lbs/Day	ACIDITY 16.0 ppm = 8.0 lbs/Day
CHLORIDES	6400 ppm = 384 lbs/Day	50 ppm =40.0 lbs/Day	46 ppm = 23.1 lbs/Day
COD	1244 ppm = 117 lbs/Day	12.4 ppm =10.6 lbs/Day	42 ppm = 20.7 lbs/Day
FREE CYANIDES	------	------	.10 ppm = .048 lbs/Day
TOTAL CYANIDES	10.8 ppm = .65 lbs/Day	.6 ppm =.58 lbs/Day	.112 ppm = .056 lbs/Da
FLUORIDES	2850 ppm = 171 lbs/Day	47 ppm =56.8 lbs/Day	5.2 ppm = 2.50 lbs/Day
SULFATES	295 ppm = 17.7 lbs/Day	170 ppm =163 lbs/Day	174 ppm = 83.6 lbs/Day
SUSPENDED SOLIDS	261 ppm = 15.7 lbs/Day	48 ppm =46.1 lbs/Day	36 ppm = 17.3 lbs/Day
CHROMIUM	.75 ppm = .045 lbs/Day	*	*
IRON	32.5 ppm = 1.95 lbs/Day	1.5 ppm =1.44 lbs/Day	.80 ppm = .38 lbs/Day
NICKEL	.45 ppm = .027 lbs/Day	*	*
POTASSIUM	6.15 ppm = .37 lbs/Day	4.91 ppm =4.72 lbs/Day	4.31 ppm = 2.07 lbs/Da
SODIUM	3150 ppm = 189 lbs/Day	109 ppm =105 lbs/Day	27.8 ppm = 13.4 lbs/Da
TIN	2500 ppm = 150 lbs/Day	12 ppm = 10.6 lbs/Day	14 ppm = 6.73 lbs/Day

* = Detectable Limit

------ = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES DATE 8/27 to 8/[illegible]

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO DEVICE	5 gpm = 7200 gpd	80 gpm = 11,200 gpd	40 gpm = 57,600 gpd
TEMPERATURE	112°F	95°F	101°F
pH	4.5	5.0	6.3
ACIDITY/ALKALINITY	[illegible] ppm = 0 lbs/Day	[illegible] ppm = 2.63 lbs/Day	[illegible] ppm = 12.5 lbs/Day
CHLORIDES	[illegible] ppm = 120 lbs/Day	35 ppm =33.6 lbs/Day	50 ppm = 27.9 lbs/ay
COD	[illegible] ppm = 60.8 lbs/Day	35 ppm =33.6 lbs/Day	50 ppm = 27.9 lbs/ ay
FREE CYANIDES	.007 ppm = .0084 lbs/Day	.0011 ppm=.0001 lbs/Day	********
TOTAL CYANIDES	4.0 ppm = .24 lbs/Day	.45 ppm =.43 lbs/Day	.05 ppm = .[illegible] lbs/Day
FLUORIDES	[illegible] ppm = 195 lbs/Day	36 ppm = 34.6 lbs/Day	2.6 ppm = 1.45 lbs/Day
SULFATES	[illegible] ppm = 17.7 lbs/Day	[illegible] ppm = 169 lbs/Day	180 ppm = 91.5 lbs/Day
SUSPENDED SOLIDS	135 ppm = 11.7 lbs/Day	45 ppm = 43.2 lbs/Day	30 ppm = 14.4 lbs/Day
CHROMIUM	.85 ppm = .051 lbs/Day	.02 ppm =.019 lbs/Day	*
IRON	35 ppm = 2.10 lbs/Day	1.3 ppm =1.25 lbs/Day	.61 ppm = .29 lbs/Day
NICKEL	.55 ppm = .033 lbs/Day	*	*
POTASSIUM	7.2? ppm = .44 lbs/Day	4.8? ppm =4.70 lbs/Day	4.6? ppm = 2.21 lbs/Day
SODIUM	[illegible] ppm = 195 lbs/ Day	71 ppm =68.1 lbs/Day	27.8 ppm = 13.4 lbs/Day
TIN	[illegible] ppm = 169 lbs/Day	45 ppm =[illegible] lbs/Day	9 ppm = 4.32 lbs/Day

* =Detectable Limit

***** = Analysis Not Performed

COUNTERCURRENT RINSING

PHASE I - CHEMICAL ANALYSES DATE 8/18/ to 8/21/

	RECOVERY TANK	RINSE WASH	HOT RINSE
FLOW TO SEWER	5 gpm = 7200 gpd	80 gpm = 115,000 gpd	40 gpm = 57,600 gpd
TEMPERATURE	112°F	95°F	104°F
pH	4.5	4.0	8.1
ACIDITY/ALKALINITY	1200 ppm = 1 lb/Day	1140 ppm = 11 lbs/Day	1140 ppm = 0.45 lbs/D
CHLORIDES	4100 ppm = 164 lbs/Day	90 ppm =86.0 lbs/Day	24 ppm = 17.2 lbs/Day
COD	1310 ppm = 70.0 lbs/Day	21 ppm =20.8 lbs/Day	14 ppm = 5.75 lbs/Day
FREE CYANIDE	.0504 ppm = 0 lbs/Day	.018 ppm =.017 lbs/Day	.0336 ppm = 0 lbs/Day
TOTAL CYANIDE	.64 ppm = 1.44 lbs/Day	.28 ppm = .27 lbs/Day	.11 ppm = .05 lbs/Day
FLUORIDES	4.900 ppm = 196 lbs/Day	51 ppm =49.0 lbs/Day	3.6 ppm = 1.72 lbs/Day
SULFATES	275 ppm = 17.7 lbs/Day	111 ppm = 107 lbs/Day	170 ppm = 81.7 lbs/Day
SUSPENDED SOLIDS	76 ppm = 6.16 lbs/Day	40 ppm =38.4 lbs/Day	5.0 ppm = 2.40 lbs/Day
CHROMIUM	.20 ppm = .016 lbs/ Day	*	*
IRON	31. ppm = 2.51 lbs/Day	1.10 ppm 1.06 lbs/Day	.53 ppm = .25 lbs/Day
NICKEL	.50 ppm = .03 lbs/Day	*	*
POTASSIUM	3.1 ppm = .25 lbs/Day	4.25 ppm =4.08 lbs/	4.34 ppm = 2.08 lbs/Da
SODIUM	4130 ppm = 331 lbs/Day	96 ppm =92.2 lbs/Day	17.8 ppm = 13.1 lbs/Da
ZINC	2780 ppm = 167 lbs/Day	42 ppm =40.4 lbs/Day	10 ppm = 4.81 lbs/Da

* = Detectable Limit

----- = Analysis Not Performed

COUNTER-CURRENT RINSING

PHASE I - CHEMICAL ANALYSIS

DATE [illegible]

	RECOVERY TANK	RINSE WASH	HOT WATER
FLOW (GPM)	[illegible]	[illegible]	[illegible]
TEMPERATURE	[illegible]	[illegible]	[illegible]
pH	[illegible]	[illegible]	[illegible]
ACIDITY/ALKALINITY	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
COD	[illegible]	[illegible]	[illegible]
TOTAL CYANIDE	[illegible]	[illegible]	[illegible]
TOTAL CHROMIUM	[illegible]	[illegible]	[illegible]
FLUORIDES	[illegible]	[illegible]	[illegible]
SULFATES	[illegible]	[illegible]	[illegible]
SUSPENDED SOLIDS	[illegible]	[illegible]	[illegible]
CHLORIDES	[illegible]	[illegible]	[illegible]
IRON	[illegible]	[illegible]	[illegible]
NICKEL	[illegible]	[illegible]	[illegible]
POTASSIUM	[illegible]	[illegible]	[illegible]
SULFIDE	[illegible]	[illegible]	[illegible]
ZINC	[illegible]	[illegible]	[illegible]

< = Detectable Limit

------ = Analysis Not Performed

COUNTERCURRENT RINSE(S)

PHASE I - CHEMICAL ANALYSES

DATE 9/3/ to 9/4/74

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO SEWER	5 gpm = 7200 gpd	80 gpm = 115,200 gpd	50 gpm = 97,600 gpd
TEMPERATURE	112°F	92°F	178°F
pH	4.3	4.3	7.0
TURBIDITY	TURBIDITY	TURBIDITY	TURBIDITY
ACIDITY/ALKALINITY	830 ppm = 49.8 lbs/Day	6.0 ppm = 5.76 lbs/Day	8.0 ppm = 3.84 lbs/Day
CHLORIDES	160 ppm = 77.3 lbs/Day	90 ppm = 69.0 lbs/Day	24 ppm = 11.5 lbs/Day
COD	814 ppm = 51.7 lbs/Day	40 ppm = 39.8 lbs/Day	41 ppm = 19.7 lbs/Day
FREE CYANIDES	******	.175 ppm = .17 lbs/Day	******
TOTAL CYANIDES	14.5 ppm = .92 lbs/Day	.181 ppm = .176 lbs/Day	.12 ppm = .058 lbs/Day
FLUORIDES	320 ppm = 175 lbs/Day	35 ppm = 33.5 lbs/Day	3.2 ppm = 1.54 lbs/Day
SULFATES	303 ppm = 18.2 lbs/Day	180 ppm = 173 lbs/Day	162 ppm = 77.8 lbs/Day
SUSPENDED SOLIDS	195 ppm = 11.7 lbs/Day	18 ppm = 17.3 lbs/Day	22 ppm = 9.61 lbs/Day
CHROMIUM	.025 ppm = .057 lbs/Day	.036 ppm = .038 lbs/Day	*
ZINC	31 ppm = 1.86 lbs/Day	1.0 ppm = .96 lbs/Day	.92 ppm = .044 lbs/Day
NICKEL	.55 ppm = .033 lbs/Day	*	*
POTASSIUM	6.7 ppm = .40 lbs/Day	4.34 ppm = 4.17 lbs/Day	3.97 ppm = 1.91 lbs/Day
SODIUM	420 ppm = 104 lbs/Day	69 ppm = 66.3 lbs/Day	27.8 ppm = 13.4 lbs/Day
TIN	2.15 ppm = .129 lbs/Day	45 ppm = 43.2 lbs/Day	12 ppm = 5.76 lbs/Day

* = Detectable Limit

****** = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES DATE 9/6/ to 9/8/

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO SEWER	5 gpm = 7200 gpd	80 gpm = 115,200 gpd	50 gpm = 57,600 gpd
TEMPERATURE	116°F	80°F	186°F
pH	6.4	6.8	8.2
ACIDITY/ALKALINITY	Acidity 24 ppm = 41.6 lbs/Day	Alkalinity 8.0 ppm = 3.76 lbs/Day	Alkalinity 12.0 ppm = 5.76 lbs/Day
CHLORIDES	1860 ppm = 116 lbs/Day	60 ppm = 57.6 lbs/Day	36 ppm = 17.3 lbs/Da
COD	884 ppm = 51.1 lbs/Day	36 ppm = 19.4 lbs/Day	36 ppm = 19.4 lbs/Da
FREE CYANIDES	======	.006 ppm = .005 lbs/Day	======
TOTAL CYANIDES	15 ppm = 7.13 lbs/Day	.08 ppm = .05 lbs/Day	.05 ppm = .13 lbs/Da
FLUORIDES	3400 ppm = 204 lbs/Day	35 ppm = 33.6 lbs/Day	2.46 ppm = 1.18 lbs/Day
SULFATES	295 ppm = 17.7 lbs/Day	186 ppm = 179 lbs/Day	166 ppm = 79.7 lbs/Day
SUSPENDED SOLIDS	210 ppm = 12.6 lbs/Day	45 ppm = 43.2 lbs/Day	25 ppm = 12.0 lbs/Day
CHROMIUM	.80 ppm = .048 lbs/Day	.03 ppm = .03 lbs/Day	*
IRON	15 ppm = 8.10 lbs/Day	1.6 ppm = 1.15 lbs/Day	.57 ppm = .27 lbs/Day
NICKEL	.50 ppm = .03 lbs/Day	*	*
POTASSIUM	6.45 ppm = .39 lbs/Day	4.27 ppm = 4.10 lbs/Day	3.77 ppm = 1.81 lbs/Day
SODIUM	3400 ppm = 204 lbs/Day	72 ppm = 69.2 lbs/Day	25.6 ppm = 12.3 lbs/Day
TIN	2735 ppm = 164 lbs/Day	30 ppm = 28.8 lbs/Day	11 ppm = 5.28 lbs/Day

* = Detectable Limit

====== = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE I - CHEMICAL ANALYSES

DATE 9/5 to 9/6/74

	RECOVERY TANK	SPRAY WASH	HOT RINSE
FLOW TO SEWER	5 gpm = 7200 gpd	80 gpm = 115,200 gpd	40 gpm = 57,600 gpd
TEMPERATURE	110°F	91°F	135°F
pH	5.3	5.5	5.9
ACIDITY/ALKALINITY	ACIDITY 440 ppm = 52.8 lbs/Day	0ppm = 0 lbs/Day	ALKALINITY 10 ppm = 7.69 lbs/Day
CHLORIDES	1300 ppm = 114 lbs/Day	75 ppm =62.4 lbs/Day	26 ppm = 12.5 lbs/Day
COD	160 ppm = 9.61 lbs/Day	8 ppm = 7.69 lbs/Day	44 ppm = 21.1 lbs/Day
FREE CYANIDES	125 ppm = .008 lbs/Day	.1 ppm = .096 lbs/Day	------
TOTAL CYANIDES	60 ppm = 3.60 lbs/Day	.3 ppm =.029 lbs/Day	.126 ppm = .061 lbs/Da
FLUORIDES	3700 ppm = 222 lbs/Day	48 ppm =46.1 lbs/Day	2.56 ppm = 1.23 lbs/Day
SULFATES	272 ppm = 16.3 lbs/Day	166 ppm = 159 lbs/Day	154 ppm = 74.0 lbs/Day
SUSPENDED SOLIDS	245 ppm = 14.7 lbs/Day	90 ppm = 86.5 lbs/Day	25 ppm = 12.0 lbs/Day
CHROMIUM	.60 ppm = .04 lbs/Day	.03 ppm = .03 lbs/Day	*
IRON	45 ppm = 2.70 lbs/Day	1.4 ppm = 1.35 lbs/Day	.61 ppm = .29 lbs/Day
NICKEL	.45 ppm = .027 lbs/Day	*	*
POTASSIUM	6.8 ppm = .41 lbs/Day	4.43 ppm = 4.26 lbs/Day	.72 ppm = 1.79 lbs/Day
SODIUM	3703 ppm = 222 lbs/Day	117 ppm = 112 lbs/Day	28.9 ppm = 13.9 lbs/Day
TIN	2700 ppm = 162 lbs/Day	64 ppm = 61.5 lbs/Day	11 ppm = 5.28 lbs/Day

* = Undetectable Limits

------ = Analysis Not Performed

COUNTERCURRENT RINSING

PERIOD III — PHASE III - CHEMICAL ANALYSIS — DATE 8/25/75

	COUNTERCURRENT TANK	ZINAY WASH	HOT RINSE
FLOW TO SEWER	0 gal/min = 0 gal/day	56,000 gal/day	42,000 gal/day
TEMPERATURE	"	87° F.	175° F.
PH	"	6.3	7.2
ACIDITY/ALKALINITY	"	Acidity 40 ppm = 3.30 lbs/Day	Alkalinity 10.0 ppm = 3.36 lbs/ay
CHLORIDES	"	83 ppm = 16.0 lbs/Day	90 ppm = 18.0 lbs/Day
COD	"	"	"
WEAK CYANIDES	"	.170 ppm = 0.35 lbs/Day	.132 ppm = .054 lbs/Day
TOTAL CYANIDES	"	.251 ppm = .05 lbs/Day	.202 ppm = .071 lbs/Day
FLUORIDES	"	.40 ppm = 8.01 lbs/Day	1.6 ppm = 1.20 lbs/Day
SULFATES	"	153.6 ppm = 30.7 lbs/Day	169.6 ppm = 57.9 lbs/ay
SUSPENDED SOLIDS	"	6.0 ppm = 3.20 lbs/Day	12.0 ppm = 4.32 lbs/Day
CHROMIUM	"	"	"
IRON	"	0.35 ppm = .07 lbs/Day	0.37 ppm = .13 lbs/Day
NICKEL	"	"	"
POTASSIUM	"	4.73 ppm = .95 lbs/Day	4.31 ppm = 1.55 lbs/Day
SODIUM	"	89 ppm = 17.8 lbs/Day	22 ppm = 7.93 lbs/Day
TIN	"	65 ppm = 13.0 lbs/Day	9 ppm = 3.24 lbs/Day

*Detectable limit
-Analysis not performed.

COUNTERCURRENT RINSING
PHASE III - CHEMICAL ANALYSES

DATE 8/29/75

	COUNTERCURRENT TANK	DIPRAX WASH	HOT RINSE
FLOW TO SEWER	0.401	84,000 gpd	42,000 gpd
TEMPERATURE	"	85° F.	177° F.
pH	"	6.1	6.9
ACIDITY/ALKALINITY	"	Acidity 10 ppm = 3.08 lbs/Day	Alkalinity 12.0 ppm = 4.12 lbs./D
CHLORIDES	"	75 ppm = 15.0 lbs/Day	300 ppm = 108 lbs./Day
COD	"	"	"
FREE CYANIDES	"	.170 ppm = .026 lbs/Day	.080 ppm = .10 lbs./Da
TOTAL CYANIDES	"	.170 ppm = .034 lbs/Day	.345 ppm = .12 lbs./Da
FLUORIDES	"	55 ppm = 8.81 lbs/Day	8.0 ppm = 2.88 lbs./Day
SULFATES	"	149.6 ppm = 29.9 lbs/Day	161.8 ppm = 51.5 lbs./D
SUSPENDED SOLIDS	"	11 ppm = 2.20 lbs/Day	22.0 ppm = 7.93 lbs./Da
CHROMIUM	"	"	"
IRON	"	0.52 ppm = .10 lbs/Day	0.51 ppm = .12 lbs./Day
NICKEL	"	"	"
POTASSIUM	"	5.62 ppm = .36 lbs/Day	4.48 ppm = 1.24 lbs./Da
SODIUM	"	87 ppm = 17.5 lbs/Day	38 ppm = 10.1 lbs./Day
TIN	"	65 ppm = 13.0 lbs/Day	16 ppm = 4.32 lbs./Day

< = Detectable Limit

" = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE III - CHEMICAL ANALYSES DATE 9/5/79

	COUNTERCURRENT TANK	SPRAY WASH	HOT RINSE
OM TO SEWER	0 gal	16,000 gal	62,025 gal
MPERATURE	-	86° F.	175° F.
	-	9.6	8.0
TIDITY/ALKALINITY	-	Alkalinity lbs/ 0.0 ppm = 0.0 lbs/Day	Alkalinity 60 ppm = 7.72 lbs/Day
LORIDES	-	16.5 ppm = 6.5 lbs/Day	18 ppm = 7.01 lbs/Day
D	-	10.6 ppm = 8.04 lbs/Day	6.8 ppm = 2.65 lbs/Day
L CYANIDES	-	.360 ppm = .078 lbs/Day	.160 ppm = .058 lbs/D
TAL CYANIDES	-	.360 ppm = .078 lbs/Day	.0046 ppm = .008 lbs/D
LUORIDES	-	.08 ppm = 3.66 lbs/Day	3.4 ppm = 1.32 lbs/Da
ULFATES	-	11.8 ppm = 23.0 lbs/Day	101 ppm = 47.6 lbs/Day
USPENDED SOLIDS	-	47 ppm = 2.63 lbs/Day	14 ppm = 5.04 lbs/Day
HROMIUM	-	*	*
OPPER	-	1.69 ppm = .30 lbs/Day	0.35 ppm = .09 lbs/Day
ICKEL	-	*	*
OTAL OIL	-	4.82 ppm = .96 lbs/Day	4.32 ppm = 1.30 lbs/Da
OLIDS	-	40 ppm = 8.41 lbs/Day	18 ppm = 6.69 lbs/Day
INC	-	25 ppm = 5.01 lbs/Day	4 ppm = 1.64 lbs/Day

* = Detectable limit
- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE III - CHEMICAL ANALYSES

DATE 9/5/75

	COUNTERCURRENT TANK	AIRAY WASH	HOT RINSE
FLOW TO SUMP	0.4 gpm	10,000 gpd	43,000 gpd
TEMPERATURE	-	85° F.	175° F.
pH	-	6.5	8.0
ACIDITY/ALKALINITY	-	0.0 ppm -	14.0 ppm = 4.36 lbs./Day
CHLORIDES	-	80 ppm =16.6 lbs/Day	61 ppm = 22.01 lbs./Day
COD	-	27.2 ppm =5.66 lbs/Day	6.8 ppm = 2.45 lbs./Day
FREE CYANIDES	-	.170 ppm = .035 lbs/Day	.09 ppm = .032 lbs./Day
TOTAL CN/NICKEL	-	.270 ppm =.056 lbs/Day	.168 ppm = .054 lbs./Day
FLUORIDES	-	5.4 ppm =10.8 lbs/Day	1.7 ppm = .61 lbs./Day
SULFATES	-	110 ppm =22.4 lbs/Day	100 ppm = 36.01 lbs./Day
SUSPENDED SOLIDS	-	75 ppm =15.6 lbs/Day	17 ppm = 6.12 lbs./Day
CHROMIUM	-	-	-
IRON	-	0.76 ppm =.15 lbs/Day	0.66 ppm = .24 lbs./Day
NICKEL	-	-	-
POTASSIUM	-	4.61 ppm =.92 lbs/Day	4.32 ppm = 1.52 lbs./Day
SODIUM	-	74 ppm =14.8 lbs/Day	14 ppm = 5.04 lbs./Day
TIN	-	52 ppm =10.4 lbs/Day	4 ppm = 1.44 lbs./Day

< = Detectable limit
- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE III - CHEMICAL ANALYSES DATE 2/11/75

	COUNTERCURRENT TANK	SPRAY WASH	HOT RINSE
OW TO FILMER	0 gal	13,800 gal	43,200 gal
MPERATURE	-	91° F.	175° F.
	-	6.6	8.1
IDITY/ALKALINITY	-	2.0 mg/l = .40 lbs/day	38 mg/l = 7.93 lbs./Day
LORIDES	-	50 mg/l = 2.76 lbs/Day	47.5 mg/l = 24.1 lbs./Day
D	-	17 mg/l = 1.38 lbs/Day	6.9 mg/l = 2.43 lbs./Day
ER CYANIDES	-	.127 mg/l = .025 lbs/day	.030 mg/l = .012 lbs./Day
TAL CYANIDES	-	.170 mg/l = .035 lbs/day	.191 mg/l = .069 lbs./Day
LORIDES	-	56.0 mg/l = 11.1 lbs/day	8.45 mg/l = .78 lbs./Day
LFATES	-	142.8 mg/l = 28.6 lbs/day	223.0 mg/l = 63.7 lbs./Da
SPENDED SOLIDS	-	47.0 mg/l = 9.34 lbs/day	20.5 mg/l = 7.21 lbs./Da
ROMIUM	-	-	-
AL	-	0.46 mg/l = .038 lbs/day	0.53 mg/l = 0.21 lbs./Da
	-	-	-
	-	6.50 mg/l = .038 lbs/day	9.80 mg/l = 1.51 lbs./Day
TASSIUM	-	50 mg/l = 9.94 lbs/Day	13 mg/l = 4.68 lbs./Day
DIUM	-	50 mg/l = 9.94 lbs/Day	6 mg/l = 2.16 lbs./Da
N			

< = Detectable Limit
- = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE III - CHEMICAL ANALYSES DATE 9/12/78

	COUNTERCURRENT TANK	SPRAY WASH	HOT RINSE
FLOW TO SEWER	1.0m³	23,816 gal	43,200 gal
TEMPERATURE	"	92° F.	180° F.
pH	"	6.6 Activity	7.0 Activity
ACIDITY/ALKALINITY	"	8.0 ppm = .40 lbs/Day	18.0 ppm = 6.62 lbs./Day
CHLORIDES	"	53 ppm = 10.3 lbs/Day	18.5 ppm = 1.67 lbs./Day
COD	"	"	"
FREE CYANIDES	"	.162 ppm = .030 lbs/Day	.082 ppm = .030 lbs./Day
TOTAL CYANIDES	"	.162 ppm = .030 lbs/Day	.0771 ppm = .0279 lbs./Day
FLUORIDES	"	48 ppm = 9.34 lbs/Day	1.84 ppm = .65 lbs./Day
SULFATES	"	146 ppm = 27.0 lbs/Day	135.6 ppm = 48.9 lbs./Day
SUSPENDED SOLIDS	"	55 ppm = 10.3 lbs/Day	38 ppm = 13.7 lbs./Day
CHROMIUM	"	"	"
IRON	"	0.50 ppm = .10 lbs/Day	0.71 ppm = .26 lbs./Day
NICKEL	"	"	"
POTASSIUM	"	4.70 ppm = .93 lbs/Day	4.25 ppm = 1.53 lbs./Day
SODIUM	"	53 ppm = 10.3 lbs/Day	12 ppm = 4.32 lbs./Day
TIN	"	125 ppm = 24.8 lbs/Day	6 ppm = 2.16 lbs./Day

* = Detectable Limit
" = Analysis Not Performed

COUNTERCURRENT RINSING
PHASE III - CHEMICAL ANALYSIS

DATE 3/18/75

	COUNTERCURRENT TANK	AIRAY WASH	HOT RINSE
...M TI [illegible]	[illegible]	[illegible]	[illegible]
TEMPERATURE	-	[illegible]	[illegible]
...I	-	[illegible]	[illegible]
...IDITY/ALKALINITY	-	[illegible]	[illegible]
CHLORIDE	-	[illegible]	[illegible]
...D	-	[illegible]	[illegible]
...NE CYANIDES	-	[illegible]	[illegible]
...TAL CYANIDES	-	[illegible]	[illegible]
FLUORIDES	-	[illegible]	[illegible]
SULFATES	-	[illegible]	[illegible]
DISSOLVED SOLIDS	-	[illegible]	[illegible]
CHROMIUM	-	-	-
IRON	-	[illegible]	[illegible]
...KEL	-	-	-
VANADIUM	-	[illegible]	[illegible]
...DIUM	-	[illegible]	[illegible]
...ER	-	[illegible]	[illegible]

* = Detectable Limit
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COUNTERCURRENT RINSING

PHASE III - CHEMICAL ANALYSES

DATE 9/19/75

	COUNTERCURRENT TANK	AIRAY WASH	HOT RINSE
FLOW	[illegible]	[illegible]	[illegible]
TEMPERATURE	[illegible]	[illegible]	[illegible]
pH	[illegible]	[illegible]	[illegible]
ACIDITY/ALKALINITY	[illegible]	[illegible]	[illegible]
CHLORIDES	[illegible]	[illegible]	[illegible]
COD	[illegible]	[illegible]	[illegible]
FREE CYANIDE	[illegible]	[illegible]	[illegible]
TOTAL CYANIDE	[illegible]	[illegible]	[illegible]
FLUORIDES	[illegible]	[illegible]	[illegible]
SULFATES	[illegible]	[illegible]	[illegible]
SUSPENDED SOLIDS	[illegible]	[illegible]	[illegible]
CHROMIUM	[illegible]	[illegible]	[illegible]
IRON	[illegible]	[illegible]	[illegible]
NICKEL	[illegible]	[illegible]	[illegible]
POTASSIUM	[illegible]	[illegible]	[illegible]
SODIUM	[illegible]	[illegible]	[illegible]
TIN	[illegible]	[illegible]	[illegible]

* <Detectable Limit

\- = Analysis Not Performed

COUNTERCURRENT RINSING

PHASE III - CHEMICAL ANALYSIS DATE 2/26/73

	COUNTERCURRENT TANK	SPRAY WASH	HOT RINSE
FLOW TO (GPM)	3 gpm	10,000 gpd	10,320 gpd
TEMPERATURE	-	85° F.	125° F.
pH	-	8.1 Alkalinity	9.3 Alkalinity
ACIDITY/ALKALINITY	-	18 ppm-3.45 lbs/Day	18.00 ppm - 3.93 lbs./Day
CHLORIDES	-	54 ppm-10.3 lbs/Day	30 ppm - 6.54 lbs./Day
COD	-	20.4 ppm-3.90 lbs/Day	6.8 ppm - 1.43 lbs./Day
HEXAVALENT CHROMIUM	-	.104 ppm-.02 lbs/Day	.066 ppm - .015 lbs./Day
TOTAL CHROMIUM	-	.104 ppm-.02 lbs/Day	.135 ppm - .066 lbs./Day
FLUORIDES	-	35.2 ppm-6.55 lbs/Day	8.0 ppm - 0.66 lbs./Day
SULFATES	-	164.8 ppm-32.2 lbs/Day	102.8 ppm - 33.7 lbs./Day
SUSPENDED SOLIDS	-	64 ppm-11.9 lbs/Day	21 ppm - 6.89 lbs./Day
CHROMIUM	-	*	*
IRON	-	0.85 ppm-.16 lbs/Day	0.93 ppm - 0.30 lbs./Day
NICKEL	-	*	*
POTASSIUM	-	4.70 ppm-0.87 lbs/Day	4.19 ppm - 1.37 lbs./Day
SODIUM	-	41 ppm-7.63 lbs/Day	14 ppm - 4.59 lbs./Day
TIN	-	40 ppm-7.45 lbs/Day	6 ppm - 1.97 lbs./Day

* Detectable Limit
- - Analysis Not Performed

COUNTERCURRENT RINSING

PHASE III - CHEMICAL ANALYSES DATE 2/21/75

	COUNTERCURRENT TANK	FINAL RINSE	HOT RINSE
FLOW	[illegible]	[illegible]	[illegible]
TEMPERATURE	[illegible]	[illegible]	[illegible]
pH	[illegible]	[illegible]	[illegible]
ACIDITY/ALKALINITY	[illegible]	[illegible]	[illegible]
CHLORIDES	[illegible]	[illegible]	[illegible]
COD	[illegible]	[illegible]	[illegible]
FREE CYANIDES	[illegible]	[illegible]	[illegible]
TOTAL CYANIDES	[illegible]	[illegible]	[illegible]
FLUORIDES	[illegible]	[illegible]	[illegible]
SULFATES	[illegible]	[illegible]	[illegible]
SUSPENDED SOLIDS	[illegible]	[illegible]	[illegible]
CHROMIUM	[illegible]	[illegible]	[illegible]
IRON	[illegible]	[illegible]	[illegible]
NICKEL	[illegible]	[illegible]	[illegible]
POTASSIUM	[illegible]	[illegible]	[illegible]
SODIUM	[illegible]	[illegible]	[illegible]
TIN	[illegible]	[illegible]	[illegible]

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(COUNTERCURRENT RINSING)

PHASE III - CHEMICAL ANALYSES DATE 10/3/73

	COUNTERCURRENT TANK	SPRAY WASH	HOT RINSE
FLOW TO SEWER	0 gpm	31,536 gpd	86,400 gpd
TEMPERATURE	-	91° F.	122° F.
pH	-	3.1	8.0
ACIDITY/ALKALINITY	-	Alkalinity 0 ppm-1.39 lbs/day	Alkalinity 26 ppm - 18.7 lbs./Day
CHLORIDES	-	18 ppm-7.36 lbs/Day	23 ppm - 20.2 lbs./Day
OIL	-	13.6 ppm-3.58 lbs/day	3.4 ppm - 2.45 lbs./Day
FLEX CLEANERS	-	.140 ppm-.037 lbs/day	.056 ppm - .036 lbs/Day
TOTAL CLEANERS	-	.160 ppm-.037 lbs/day	.073 ppm - .053 lbs./Day
FLUORIDES	-	13.0 ppm-3.44 lbs/day	1.2 ppm - 0.86 lbs./Day
OIL-RATED	-	77.2 ppm-20.5 lbs/day	91 ppm - 65.3 lbs./Day
SUSPENDED SOLIDS	-	54 ppm-14.2 lbs/Day	18 ppm - 13.0 lbs./Day
CHROMIUM	-	*	*
IRON	-	0.58 ppm-.15 lbs/Day	0.84 ppm - .61 lbs./Day
NICKEL	-	*	*
MOLYBDENUM	-	4.63 ppm-1.22 lbs/day	4.10 ppm - 3.03 lbs./Day
BORIDES	-	35 ppm-9.61 lbs/Day	11 ppm - 7.95 lbs./Day
ZINC	-	21 ppm-5.53 lbs/Day	5 ppm - 3.60 lbs./Day

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